

REMARKS

Claims 1-22, 37 and 38 are rejected. Claims 23-36 are withdrawn from consideration. Claims 1-38 are presently pending in the application. Favorable reconsideration of the application in view of the following remarks is respectfully requested.

Rejection of Claim 38 under 35 USC § 112:

The Examiner has rejected Claim 38 is rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement, indicating that the claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention, as the Examiner was unable to find support in the original claims or specification for the limitation that the vinyl latex polymer be anionic.

Example 4, pg. 17, line 26 of the application makes specific mention of an anionic latex, Morcryl 132 vinyl latex. Information contained in any one of the specification, claims or drawings of the application as filed may be added to any other part of the application without introducing new matter. MPEP 2163.06

The anionic nature of morcryl is an inherent characteristic of the chemical compound bearing the tradename morcryl. This characteristic, resulting from the known chemical structure of the compound would be obvious to one of ordinary skill in the chemical formulating art related to ink recording elements. According to MPEP2163.07(a) Inherent Function, Theory, or Advantage, by disclosing in a patent application a device that inherently performs a function or has a property, operates according to a theory or has an advantage, a patent application necessarily discloses that function, theory or advantage, even though it says nothing explicit concerning it. The application may later be amended to recite the function, theory or advantage without introducing prohibited new matter. In re Reynolds, 443 F.2d 384, 170 USPQ 94 (CCPA 1971); In re Smythe, 480 F. 2d 1376, 178 USPQ 279 (CCPA 1973).

The specification also clearly discloses anionic polyurethane dispersions (pg. 7, line 4, pg. 8, lines 24-28 and pg. 17, Examples 2, 3). The specification also discloses vinyl latex polymers (pg. 6, lines3-5) and Example 4 specifically discloses an anionic vinyl latex polymer. It would be clear to one of

ordinary skill in the art that, is anionic polyurethane dispersions are preferred, other anionic components, such as vinyl latex polymers, would be preferred. It would be clear to one of ordinary skill in the formulary arts from the exemplified formulations of the Examples, that anionic materials are necessary, in order to produce non-agglomerated, coatable materials.

According to MPEP 2164.05(a): The specification need not disclose what is well-known to those skilled in the art and preferably omits that which is well-known to those skilled and already available to the public. In re Buchner, 929 F.2d 660, 661, 18 USPQ2d 1331, 1332 (Fed. Cir. 1991); Hybritech, Inc. v. Monoclonal Antibodies, Inc., 802 F.2d 1367, 1384, 231 USPQ 81, 94 (Fed. Cir. 1986), cert. denied, 480 U.S. 947 (1987); and Lindemann Maschinenfabrik GMBH v. American Hoist & Derrick Co., 730 F.2d 1452, 1463, 221 USPQ 481, 489 (Fed. Cir. 1984). "That what appellants claim as patentable to them is less than what they describe as their invention is not conclusive if their specification also reasonably describes that which they do claim. Inventions are constantly made which turn out not to be patentable, and applicants frequently discover during the course of prosecution that only a part of what they invented and originally claimed is patentable." In re Wertheim, 541 F.2d 257, 263 (CCPA, 1976). "Since the patent law provides for the amendment during prosecution of claims, as well as the specification supporting claims, 35 USC 132, it is clear that the reference to "particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention" in the second paragraph of 35 USC 112 does not prohibit the applicant from changing what he "regards as his invention" (i.e., the subject matter on which he seeks patent protection) during the pendency of his application." Cf. In re Brower, 58 CCPA 724, [728] 433 F.2d 813, 817, 167 USPQ 684, 687 (1970). In the present application, the Examples clearly teach anionic polyurethane dispersions as well as anionic vinyl latex polymers. The Applicants therefore believe that Claim 38 does not introduce new matter.

SUMMARY

To summarize the key points of the following remarks:

The term "lamine adhesion" and the term "adhesion" have accepted and different meanings in the art, as evidenced by ASTM test methods.

The language of the experimental examples have been clarified to support Example 1 and Control Example 5, one utilizing derivatized PVA and the control utilizing non-derivatized PVA, and the copies of the documentation relating to the original experiments as well as copies of a new set of experiments duplicating the originals are included.

Based on the teachings of the prior art, one would not expect a single layer to affect the adhesion of other layers in a multi-layer with which it is not in contact, as well as the adhesion of the whole pack to the substrate, which substrate is also not adjacent to the laminate adhesion promoting layer.

Rejection of Claims 1-3, 9, 11, 12, 15, 21, 22 and 37 Under 35 U.S.C. §102(b):

The Examiner has rejected Claims 1-3, 9, 11, 12, 15, 21, 22 and 37 are rejected under 35 U.S.C. 102(b) as described in the Office communication dated 9/28/2004. The Examiner indicates that the term "laminate adhesion promoting absorbing hydrophilic overcoat polymer latex" has no specific meaning in the art and is largely a series of functions assigned to the layer and, since the top layer of the reference would have each of these properties to some degree, the claimed layer reads on the top layer of the reference.

Kawano relates to an ink jet recording sheet comprising a substrate and an ink receptive layer disposed on said substrate, said ink receptive layer containing a pigment and a binder as its main components, the improvement comprising said ink receptive layer being an aqueous composition containing a pigment and an amphoteric latex as its main components, which absorbs water-base ink well, gives high-grade images, and ensures excellent water resistance of printed images. Kawano fails to disclose difficulties with laminate adhesion and fails to disclose the use of a laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) having at least one hydroxyl group replaced by ether or ester groupings.

The present invention comprises an ink recording element comprising a support having a hydrophilic absorbing layer and a laminate adhesion promoting absorbing hydrophilic overcoat polymer layer containing derivatized poly(vinyl alcohol) having at least one hydroxyl group replaced by ether or ester groupings which provides better laminate adhesion than the elements of the prior art, while maintaining other properties such as excellent image quality, no banding, bleeding, coalescence, or cracking in inked areas,

absorption of large amounts of ink, quick drying to avoid blocking, high optical densities in the printed areas, freedom from differential gloss and high levels of image fastness.

A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. The reference to Kawano fails to teach or suggest a laminate adhesion promoting overcoat layer. As a result, Kawano fails to anticipate the presently claimed invention.

In performing this examination, the Examiner has been correctly defining the phrase "laminate adhesion" as referring to the adhesion of a coating as part of a laminate. However, there are other aspects to laminate adhesion, as is illustrated by ASTM Test Method F2226-03, Section 4.6 and FIG.2 on pg. 2, provided as Attachment A. The term "laminate adhesion" also includes adhesion of the laminate to the underlying substrate, as well as the adhesion of each layer to an adjacent layer and adhesion of all the laminate layers together. The cited prior art contains a different term, that is, simple "adhesion", which refers to the adhesion between a layer and the adjacent layer, that is, the substrate. Simple adhesion is understood to refer to the relationship between the substrate and layers applied directly to the substrate. See ASTM Test Method D3359-02, especially the Appendix at X1.3.2., provided as Attachment B. The inventive element presently claimed has an adhesion promoting overcoat layer. The use of this layer improves the adhesion of all the layers, both to adjacent layers and to the substrate. In laminate adhesion as presently used, the substrate is not in contact with the adhesion promoting overcoat layer. In the prior art, enhanced adhesion involves an adjacent layer. Therefore, the definitions of the terms "adhesion" and "laminate adhesion" are not synonymous and the reference cannot anticipate the present claims.

Further, the cited prior art indicates that the term "adhesion" is specific to the interaction between the substrate and the coated layer, as evidenced by Kawano, col. 1 line 65 – col. 2, line 2 ("If a synthetic paper or a plastic film is used as a substrate or a Latex is used as a binder, then the ink jet recording sheet will have the disadvantages that the adhesion between the ink receptive layer and the substrate is weak and ink is not dried or absorbed well."), Kawano, col. 4 lines 47-50 ("The amphoteric latex used in the present invention displays its

characteristic features as in the following when a synthetic resin material having a strong barrier property such as a plastic film and a synthetic paper is used as a substrate: The ink receptive layer matches the substrate better, and as a result strong adhesion is obtained.”), Kawano, col. 8 lines 9-12 (“Second, in said bottom layer, if the amount of the amphoteric latex is larger than the amount of the water-soluble high polymer, then the adhesion of the ink receptive layer to the substrate is increased.”), Kawano, col. 8 lines 31-35 (“If the amount of the amphoteric latex is below 40 parts by weight or smaller than the amount of the water-soluble high polymer, then the adhesion of the ink receptive layer to the substrate is unfavorably lowered.”), Kawano, col. 8, lines 44-48 (“In the bottom layer of the ink receptive layer, if the amount of the water-soluble high polymer is above 30 parts by weight per 100 parts by weight of pigment, then the adhesion of the ink receptive layer to the substrate is unfavorably lowered...”), Ueda, pg. 27, paragraph [0090] (“The layer provided on one or each side of the support may contain a matting agent in an amount of 0.005 to 0.1 g/m² in order to minimize adhesion failure such as blocking.”), and Tomizawa, col. 4 lines 23-29 (“If desired in order to improve the adhesion between the substrate surface and the ink-receptive layer thereon, it is optional to subject the substrate surface to a treatment having a priming effect such as a corona discharge treatment, ozone treatment and plasma treatment as well as to a coating treatment to form an undercoating layer of a saturated polyester resin or urethane resin having a thickness in the range, for example, from 0.5 to 5 μ m.”)

Rejection Of Claims 1-3, 7-15,19-22 and 37 Under 35 U.S.C. §103(a):

The Examiner has rejected Claims 1-3, 7-15,19-22 and 37 under 35 U.S.C. 103(a) as being unpatentable over Kawano et al. (5,478,631) for reasons of record and as described in the Office communication dated 9/28/2004.

To establish a prima facie case of obviousness requires, first, there must be some suggestion or motivation, either in the references themselves, or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art references (or references when combines) must teach or suggest all the claim limitations. Kawano fails to teach, disclose or suggest a laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion. Kawano

also fails to mention the property of laminate adhesion. Therefore, the reference fails to provide any motivation to modify the reference to produce the laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion as presently claimed. The reference also fails to provide any likelihood of obtaining improved laminate adhesion by using a derivatized poly (vinyl alcohol) as presently claimed in an overcoat layer. One would not expect a layer to improve the adhesion properties of the whole coated laminate pack, especially regarding the adhesion of other layers to each other, which are not in contact with the overcoat layer. Finally, since the reference fails to mention laminate adhesion or the use of derivatized poly (vinyl alcohol) in an overcoat layer to improve laminate adhesion, the reference fails to teach, disclose or suggest the limitations of the invention as presently claimed.

Even assuming a prima facie case of obviousness has been made, the present invention provides surprising results. Kawano teaches the use of poly(vinyl alcohol) (col. 6, lines 1-15). The present invention, Example 1, which has an overcoat containing derivatized PVA demonstrates excellent laminate adhesion (Table 2, page 18) as compared to Example 5, having an overcoat layer containing non-derivatized PVA, as well as Example 6, having an overcoat containing PVA and polyethyleneoxide copolymer.

The Examiner has indicated concern with the Examples, indicating that the examples do not vary only the polyvinyl alcohol, as, in each of examples 1-4, the derivatized polyvinyl alcohol is used along with another material. However, Examples 1 and Control Example 5 are identical, except for the variation in poly(vinyl alcohol), derivatized vs. non-derivatized. Example 1, as copied from the as-filed specification states:

“A polyethylene resin coated paper was treated by corona discharge and coated by means of an extrusion/slide hopper with a 10% gelatin solution in water, (succinylated pigskin gelatin, Kind & Knox Gelatine Co.), and 0.6% 12 micron polystyrene beads, dry coverage of about 8.5 microns and an inner layer of 5% solution of Elvanol 52-22 poly(vinyl alcohol) (DuPont) and a 30% dispersion of Witcobond 232 polyurethane (Witco Corp), where the poly(vinyl alcohol) (PVA) and polyurethane dispersion (PUD) were mixed in a 77:23 ratio by weight at a dry coverage of 1.5 microns. An overcoat layer consisting of a 2%

solution of Z-320 acetoactylated poly(vinyl alcohol) (Nippon Gohsei) and APG 325N (Cognis) and Surfactant 10G (Arch Chemical) surfactants in a ratio by weight of 96.9/2.4/0.7 was coated over the gelatin and poly(vinyl alcohol)/polyurethane layers at a dry coverage of 1 micron. The coatings were dried thoroughly by forced air heat after application of the coating solutions.”

Control Example 1, copied from the as-filed specification states:

As in example 1 except that the overcoat layer consisted of hydroxyethyl cellulose (HEC QP 300, Dow).

Using the exact terms of Example 1, Control Example 1 would appear as follows:

“A polyethylene resin coated paper was treated by corona discharge and coated by means of an extrusion/slide hopper with a 10% gelatin solution in water, (succinylated pigskin gelatin, Kind & Knox Gelatine Co.), and 0.6% 12 micron polystyrene beads, dry coverage of about 8.5 microns and an inner layer of 5% solution of Elvanol 52-22 poly(vinyl alcohol) (DuPont) and a 30% dispersion of Witcobond 232 polyurethane (Witco Corp), where the poly(vinyl alcohol) (PVA) and polyurethane dispersion (PUD) were mixed in a 77:23 ratio by weight at a dry coverage of 1.5 microns. An overcoat layer consisting of a 2% solution of hydroxyethyl cellulose (HEC QP 300, Dow) and APG 325N (Cognis) and Surfactant 10G (Arch Chemical) surfactants in a ratio by weight of 96.9/2.4/0.7 was coated over the gelatin and poly(vinyl alcohol)/polyurethane layers at a dry coverage of 1 micron. The coatings were dried thoroughly by forced air heat after application of the coating solutions.”

Control Example 5, copied from the as-filed specification states:

As in control example 1 except that the overcoat layer consisted of a non-acetoactylated poly(vinyl alcohol) (GH-23, Nippon Gohsei).

Using the exact terms of Example 1 and Control Example 1, Example 5 would appear as follows:

“A polyethylene resin coated paper was treated by corona discharge and coated by means of an extrusion/slide hopper with a 10% gelatin solution in water, (succinylated pigskin gelatin, Kind & Knox Gelatine Co.), and 0.6% 12 micron polystyrene beads, dry coverage of

about 8.5 microns and an inner layer of 5% solution of Elvanol 52-22 poly(vinyl alcohol) (DuPont) and a 30% dispersion of Witcobond 232 polyurethane (Witco Corp), where the poly(vinyl alcohol) (PVA) and polyurethane dispersion (PUD) were mixed in a 77:23 ratio by weight at a dry coverage of 1.5 microns. An overcoat layer consisting of a 2% solution of As in control example 1 except that the overcoat layer consisted of a non-acetoacetylated poly(vinyl alcohol) (GH-23, Nippon Gohsei) and APG 325N (Cognis) and Surfactant 10G (Arch Chemical) surfactants in a ratio by weight of 96.9/2.4/0.7 was coated over the gelatin and poly(vinyl alcohol)/polyurethane layers at a dry coverage of 1 micron. The coatings were dried thoroughly by forced air heat after application of the coating solutions.”

Copies of the original laboratory notebook pages are included as Attachment C-1 (Layer / Melt Compositions), Attachment C-2 (Coating Compositions – Inventive), Attachment C-3 (Coating Compositions – Control) and Attachment C-4 (Physical Evaluation). In addition, the inventor has duplicated the experimental work, including a set of examples which do not include surfactant. This data is included as Attachment D-1 (Data / Results), Attachment D-2 (Layer / Melt Compositions) and Attachment D-3 (Coating Compositions) and indicates that the peel force needed for Inventive Examples 1 and 5 is significantly greater than the peel force for Control Examples 5 and 11, indicating greatly improved laminate adhesion of the inventive sample over the control. In addition, Examples 5-8 and Control Examples 7-12 illustrate that the surfactants utilized in the overcoat layer do not affect laminate adhesion, only coatability of the laminate.

The Examiner indicates that, while Kawano et al. does not refer to any layer in this manner, the term "laminate adhesion promoting absorbing hydrophilic" is merely a list of properties that are attributed to the overcoat layer in the instant claims and the top layer of Kawano et al. will possess these properties to some degree because the layers adhere in a laminate, are hydrophilic, are made of polymers and provide an overcoat. As discussed above, laminate adhesion relates to more than just the overcoat layer itself. The overcoat layer promotes the adhesion to the adjacent layer, but applies to enhancing the adhesion of all the layers in the laminate – to each adjacent layer, to non-adjacent layers, as

well as the whole laminate to the substrate. Kawano makes no disclosure that would lead one of ordinary skill in the art to expect a distant, non-adjacent layer to improve the adhesion of the whole structure.

Therefore, since the reference fails to provide any suggestion or motivation to modify the reference, fails to provide a reasonable expectation of success, fails to teach or suggest all the claim limitations, and in the light of surprising results, the Applicants respectfully request the Examiner to reconsider and withdraw the rejection.

Rejection Of Claim 38 Under 35 U.S.C. §103(a):

The Examiner has rejected Claim 38 is rejected under 35 U.S.C. 103(a) as being unpatentable over Kawano et al. (5,478,631) for reasons of record and further in view of applicants' admission of pages 8-9 of the specification for reasons given below. Applicants admit that polyurethane that may be used in their invention may be Witcobond ® W-232, a commercially available polyurethane dispersion. Based upon the disclosure of the reference that a polyurethane may be included in the disclosed medium, it would have been obvious to one of ordinary skill in the art to include a commercially available polyurethane that is capable of forming part of an aqueous coating composition.

As discussed above, Kawano fails to teach or suggest the improvements in laminate adhesion achievable with the use of acetoacetylated poly(vinyl alcohol) in combination with an anionic vinyl latex polymer or an anionic polyurethane dispersion in the overcoat layer. Kawano also fails to disclose the use of anionic materials, utilizing an amphoteric latex instead. For the reasons discussed above, the Applicants believe that the use of acetoacetylated poly(vinyl alcohol) is non-obvious, as is the combination with anionic vinyl latex polymer or an anionic polyurethane dispersion.

Rejection Of Claims 1, 2, 4-6 and 37 Under 35 U.S.C. §103(a):

The Examiner has rejected Claims 1, 2, 4-6 and 37 under 35 U.S.C. 103(a) as being unpatentable over Kawano et al. as applied to claims 1-3, 7-15 and 19-22 above, and further in view of Tomizawa et al. (6,224,971) for reasons of record and as described in the Office communication dated 9/28/2004.

Tomizawa discloses novel ink-jet recording sheets having high water resistance, excellent transparency of the ink-receptive layer, ink absorptivity and color developability as well as the advantages of absence of

surface tackiness and blocking and a liquid coating composition for forming the ink-receptive coating layer of the recording sheet. The liquid coating composition comprises, as a uniform blend in an aqueous medium, an acetoacetylated polyvinyl alcohol, a polyvinylpyrrolidone resin, and an acidic aqueous dispersion of a colloidal silica, each in a specified weight proportion. Tomizawa fails to disclose laminate adhesion or the use of a laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) having at least one hydroxyl group replaced by ether or ester groupings. Tomizawa deals with a single coated layer on a substrate, which would suffer from adhesion failure, but not laminate adhesion failure due to the absence of a multiple layer laminate.

To establish a prima facie case of obviousness requires, first, there must be some suggestion or motivation, either in the references themselves, or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art references (or references when combines) must teach or suggest all the claim limitations. Kawano and Tomizawa fail to teach, disclose or suggest a laminate adhesion promoting absorbing hydrophilic overcoat polymer layer containing derivatized poly(vinyl alcohol) having at least one hydroxyl group replaced by ether or ester groupings which provides better laminate adhesion. Tomizawa and Kawano also fail to mention the property of laminate adhesion. In fact, Tomizawa fails to disclose a laminate structure, disclosing instead a single coated layer on a substrate. Therefore, the references fail to provide any motivation to modify the reference to produce the laminate adhesion promoting absorbing hydrophilic overcoat polymer layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion as presently claimed. The Examiner notes that it would have been obvious to one of ordinary skill in the art to determine the particular polyvinyl alcohol from the commercially available series of polymers identified by the prior art as useful in ink jet recording media applications in order to achieve the goals set forth by the primary reference. However, Kawano fails to teach the goal of improved laminate adhesion. The references also fail to provide any likelihood of obtaining improved laminate adhesion by using a derivatized poly (vinyl alcohol) as presently claimed. Finally, since the references fail to mention laminate adhesion or the use of derivatized poly (vinyl alcohol) in an overcoat layer to

improve laminate adhesion, the reference fails to teach, disclose or suggest the limitations of the invention as presently claimed.

Also, as discussed above, the present invention provides evidence of surprising results. As discussed above, laminate adhesion relates to more than just the overcoat layer itself. The overcoat layer promotes the adhesion to the adjacent layer, but applies to enhancing the adhesion of all the layers in the laminate – to each adjacent layer, to non-adjacent layers, as well as the whole laminate to the substrate. One would not expect a layer to improve the adhesion properties of the whole coated laminate pack, especially regarding the adhesion of other layers to each other, which are not in contact with the overcoat layer. Kawano makes no disclosure that would lead one of ordinary skill in the art to expect a distant, non-adjacent layer to improve the adhesion of the whole structure and Tomizawa deals with only a single layer.

Therefore, since the references fail to suggest or motivate one to modify or combine the references, fail to provide a reasonable expectation of success, fail to teach or suggest all the claim limitations, and in the light of surprising results, the Applicants respectfully request the Examiner to reconsider and withdraw the rejection.

Rejection Of Claims 1 and 15-18 Under 35 U.S.C. §103(a):

The Examiner has rejected Claims 1 and 15-18 under 35 U.S.C. 103(a) as being unpatentable over Kawano et al. as applied to claims 1-3, 7-15, 19-22 above, and further in view of Ueda et al. (EP 791,475) for reasons of record and as described in the Office communication dated 9/28/2004.

Ueda discloses a recording sheet for ink-jet recording, which comprises a support, and provided thereon, an ink receiving layer, wherein the ink receiving layer contains a water soluble polymer, a polymer latex and gelatin, to provide an excellent image under various conditions and an excellent transportability. Ueda fails to disclose laminate adhesion as a problem, and fails to disclose the use of derivatized poly(vinyl alcohol).

To establish a prima facie case of obviousness requires, first, there must be some suggestion or motivation, either in the references themselves, or in the knowledge generally available to one of ordinary skill in the art, to modify the reference or to combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art references (or references when

combines) must teach or suggest all the claim limitations. Kawano and Ueda fail to teach, disclose or suggest a laminate adhesion promoting absorbing hydrophilic overcoat polymer layer containing derivatized poly(vinyl alcohol) having at least one hydroxyl group replaced by ether or ester groupings which provides better laminate adhesion. Ueda and Kawano also fail to mention the property of laminate adhesion. Therefore, the references fail to provide any motivation to modify the reference to produce the laminate adhesion promoting absorbing hydrophilic overcoat polymer layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion as presently claimed. The references also fail to provide any likelihood of obtaining improved laminate adhesion by using a derivatized poly (vinyl alcohol) as presently claimed. Finally, since the references fail to mention laminate adhesion or the use of derivatized poly (vinyl alcohol) in an overcoat layer to improve laminate adhesion, the reference fails to teach, disclose or suggest the limitations of the invention as presently claimed.

Also, as discussed above, the present invention provides evidence of surprising results. As discussed above, laminate adhesion relates to more than just the overcoat layer itself. The overcoat layer promotes the adhesion to the adjacent layer, but applies to enhancing the adhesion of all the layers in the laminate – to each adjacent layer, to non-adjacent layers, as well as the whole laminate to the substrate. One would not expect a layer to improve the adhesion properties of the whole coated laminate pack, especially regarding the adhesion of other layers to each other, which are not in contact with the overcoat layer. Kawano makes no disclosure that would lead one of ordinary skill in the art to expect a distant, non-adjacent layer to improve the adhesion of the whole structure and Tomizawa deals with only a single layer.

Therefore, since the references fail to suggest or motivate one to modify or combine the references, fail to provide a reasonable expectation of success, fail to teach or suggest all the claim limitations, and in the light of surprising results, the Applicants respectfully request the Examiner to reconsider and withdraw the rejection.

Rejection of Claims 1, 2, 7, 8, 21, 22 and 37 under 35 USC § 102(e):

The Examiner has rejected Claims 1, 2, 7, 8, 21, 22 and 37 under 35 U.S.C. 102(e) as being anticipated by Niu et al. (6,599,593), as the reference discloses an ink-receiving medium that may be used as an ink jet recording

medium comprising a substrate, a porous medial layer, and a non-porous top layer on the surface thereof, in which the top layer may be a combination of an acetoacetylated polyvinyl alcohol, a polyurethane resin dispersion and an epichlorohydrin containing polyamide, the medial layer may be pigment and polyvinyl alcohol, the layer may also contain a latex resin, an additional layer may be present between the top layer and the medial layer, and the epichlorohydrin containing polyamide acts as a mordant.

Niu discloses an ink-receiving media print media products having multi-functional capabilities including (A) minimal drying time; (B) improved gloss-control which allows the production of high-gloss or semi-gloss products as desired; and (C) the ability to generate high-definition and light- fast images. A first embodiment (high-gloss) employs a substrate (e.g. polyethylene-coated paper) having a porous medial layer thereon containing a pigment (e.g. silica) and a binder (e.g. polyvinyl alcohol). A non-porous top layer is placed on the medial layer which optimally consists entirely of one or more binders (excluding pigments, etc.) A second embodiment (semi-gloss) employs a comparable substrate and medial layer with a top layer similar to that described above, although at least one pigment (e.g. silica) is employed therein at a level not exceeding about 10% by weight. Niu fails to mention a laminate adhesion promoting overcoat layer.

A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. The reference to Niu fails to teach or suggest a laminate adhesion promoting overcoat layer. As a result, Niu fails to anticipate the presently claimed invention. Neither does Niu inherently disclose such a property, as Niu includes the use of carboxycellulose materials as the binder in the overcoat layer, which compounds have been used by the Applicant as control samples. As a result Niu fails to anticipate the laminate adhesion promoting overcoat layer as presently claimed.

Rejection Of Claims 1, 2, 9-14, 19-22, 37 and 38 Under 35 U.S.C. §103(a):

The Examiner has rejected Claims 1, 2, 9-14, 19-22, 37 and 38 under 35 U.S.C. 103(a) as being unpatentable over Niu et al. as above.

To establish a prima facie case of obviousness requires, first, there must be some suggestion or motivation, either in the references themselves, or in the knowledge generally available to one of ordinary skill in the art, to modify the

reference or to combine reference teachings. Second, there must be a reasonable expectation of success. Finally, the prior art references (or references when combines) must teach or suggest all the claim limitations. Niu fails to teach, disclose or suggest a laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion. Niu also fails to mention the property of laminate adhesion. Therefore, the reference fails to provide any motivation to modify the reference to produce the laminate adhesion promoting overcoat layer containing derivatized poly(vinyl alcohol) which provides better laminate adhesion as presently claimed. The reference also fails to provide any likelihood of obtaining improved laminate adhesion by using a derivatized poly (vinyl alcohol) as presently claimed in an overcoat layer. One would not expect a layer to improve the adhesion properties of the whole coated laminate pack, especially regarding the adhesion of other layers to each other, which are not in contact with the overcoat layer. In addition, Niu indicates that secondary binders other than derivatized PVA and polyurethane may be used. At least one of these other secondary binders, carboxycellulose, has been utilized as Control Example 4, over which the presently claimed composition demonstrates surprisingly approved adhesion. At best, Niu provides a list of materials that would be obvious to try, some of which have been found to not promote laminate adhesion. Finally, since the reference fails to mention laminate adhesion or the use of derivatized poly (vinyl alcohol) in an overcoat layer to improve laminate adhesion, the reference fails to teach, disclose or suggest the limitations of the invention as presently claimed.

Rejection Of Claim 38 Under 35 U.S.C. §103(a):

The Examiner has rejected Claim 38 is rejected under 35 U.S.C. 103(a) as being unpatentable over Niu et al. (6,599,593) for reasons of record and further in view of applicants' admission of pages 8-9 of the specification for reasons given below. Applicants admit that polyurethane that may be used in their invention may be Witcobond ® W-232, a commercially available polyurethane dispersion. Based upon the disclosure of the reference that a polyurethane dispersion may be included in the disclosed medium, it would have been obvious to one of ordinary skill in the art to include a commercially available polyurethane that is capable of forming part of an aqueous coating composition.

As discussed above, Niu fails to teach or suggest the improvements in laminate adhesion achievable with the use of acetoacetylated poly(vinyl alcohol) in combination with an anionic vinyl latex polymer or an anionic polyurethane dispersion in the overcoat layer. Niu also fails to disclose the use of anionic materials, utilizing an amphoteric latex instead. For the reasons discussed above, the Applicants believe that the use of acetoacetylated poly(vinyl alcohol) is non-obvious, as is the combination with anionic vinyl latex polymer or an anionic polyurethane dispersion.

Rejection Of Claims 1, 2 and 4-6 Under 35 U.S.C. §103(a):

The Examiner has rejected Claims 1, 2 and 4-6 under 35 U.S.C. 103(a) as being unpatentable over Niu et al. (6,599,593) as applied to claims 1, 2, 7-14, 19-22, 37 and 36 above, and further in view of Tomizawa et al. (6,224,971), indicating that the secondary reference discloses an ink jet recording sheet including acetoacetylated polyvinyl alcohol, the primary reference does not disclose the properties of the disclosed acetoacetylated polyvinyl alcohol used therein, therefore, it is appropriate to look elsewhere in the same art area for a particular acetoacetylated polyvinyl alcohol to use in the invention of the primary reference.

As discussed above, the Applicants believe that the present invention is not obvious in light of either the reference to Niu or Tomizawa. The motivation to combine references must come from statements in the prior art, the knowledge of one of ordinary skill in the art or from the nature of the problem to be solved. In re Kotzab, 217 F.3d 1365, 1370 (Fed Cir. 2000). Niu fails to mention laminate adhesion, providing no motivation to look elsewhere. The goals set out in Niu do not include improving laminate adhesion. Tomizawa deals with a single layer coating applied to a substrate and fails to teach anything relating to laminate adhesion.

Rejection Of Claims 1 and 15-18 Under 35 U.S.C. §103(a):

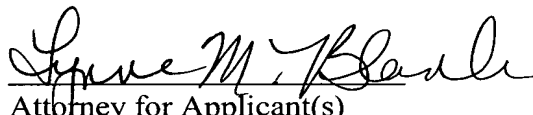
The Examiner has rejected Claims 1 and 15-18 under 35 U.S.C. 103(a) as being unpatentable over Niu et al. (6,599,593) as applied to claims 1, 2, 7-14, 19-22, 37 and 38 above, and further in view of Ueda et al. (EP 791,475).

As discussed above, the Applicants believe that the present invention is not obvious in light of either the reference to Niu or Ueda. The combination of Niu and Ueda would fail to produce the presently claimed

derivatized poly (vinyl alcohol) used in an overcoat layer to improve laminate adhesion. At best, Niu provides a list of materials that would be obvious to try in an overcoat layer, some of which have been found to not promote laminate adhesion in combination with a gelatinous layer. Neither Niu nor Ueda disclose laminate adhesion. Finally, since the references fail to mention laminate adhesion or the use of derivatized poly (vinyl alcohol) in an overcoat layer to improve laminate adhesion, the references fail to teach, disclose or suggest the limitations of the invention as presently claimed.

It is believed that the foregoing is a complete response to the Office Action and that the claims are in condition for allowance. Favorable reconsideration and early passage to issue is therefore earnestly solicited.

Respectfully submitted,


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If the Examiner is unable to reach the Applicant(s) Attorney at the telephone number provided, the Examiner is requested to communicate with Eastman Kodak Company Patent Operations at (585) 477-4656.



Standard Practice for Determining the Adhesion of Prints and Laminating Films¹

This standard is issued under the fixed designation F 2226; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for printing a document, applying a laminating film and subsequently delaminating. The laminate film is used for encapsulating and mounting prints to preserve them in office and outdoor environments.

1.2 This practice describes procedures for determining the adhesion strength of lamination film when it is applied to black and white and color prints produced by printers, copiers and other reprographic devices.

1.3 This practice can be used to test different laminates with a given set of inks and media or it can be used to evaluate inks and media with a given laminate.

1.4 This practice specifies size of specimens and defines conditions for measurement of peel adhesion at a 180° angle and delamination speed.

1.5 This practice is applicable to constructions where the substrate surface is subject to failure under peel conditions.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 Definitions:

2.1.1 *high temperature lamination*—lamination at temperature not lower than 80°C (180°F). The maximum applied temperature is 115 to 121°C (240 to 250°F).

2.1.2 *lamination film*—plastic film having an adhesive layer on one side. The film can be glossy, semi-glossy, or matte and contain additives modifying its optical properties. The film usually has a thickness between 25 to 250 μ (1 to 10 mils).

2.1.3 *low temperature lamination*—lamination at room temperature with applied pressure mostly to mounting board using a pressure-sensitive adhesive.

2.1.4 *mounting board*—paperboard, plastic board, or any supporting board, which could be used to display prints.

2.1.5 *printed media*—recording elements used by printers to receive inks or toners. The substrate may be paper, plastic, canvas, fabric, or other ink receptive material. The substrate may, or not, be coated with an ink receptive layer(s).

2.2 Definitions of Terms Specific to This Standard:

2.2.1 *adhesion strength of a laminate*—load per unit necessary to remove the laminate from a prescribed surface when measured in accordance with this test method.

2.2.2 *delaminating/debonding*—separation of a laminating film from a printed media.

2.2.3 *kinetic peak*—load per unit presenting the maximum force that occurs during the average time during the peel test. See Fig. 1.

2.2.4 *peel or stripping strength*—the average load per unit width of sample required to separate or peel the laminate from the printed media at the adhered interface at a separation angle of approximately 180° and at a separation rate of preferably 308 mm (12 in.) per minute. It is expressed in Newtons per meter width, grams per inch width, or ounces per inch width.

2.2.5 *root-mean-square (RMS)*—a mathematical treatment of the force data intended to qualify the extent to which the value deviates about its average value.

2.2.6 *static peak*—load per unit presenting the maximum force that occurs during the delay time before starting the peel test. See Fig. 1.

2.2.7 *valley*—load per unit presenting minimum force during the average time under kinetic conditions. See Fig. 1.

3. Summary of Practice

3.1 Coated or uncoated substrate is printed under standard conditions using four primary, three secondary colors and composite black. Eight color strips 2.54 mm wide and one unprinted are generated.

3.2 The prints having eight color strips are conditioned (preferably for 24 h) and subsequently laminated.

3.3 The laminated prints are conditioned (preferably for 24 h), cut into 25.4-mm strips and subsequently delaminated.

3.4 Comparative studies require testing under well-defined conditions.

4. Significance and Use

4.1 The image life of printed media displayed in both typical office and outdoor environments can be extended by lamination or encapsulation. While natural aging is the most reliable method of assessing lamination adhesion, the length of

¹ This practice is under the jurisdiction of ASTM Committee F05 on Business Imaging Products and is the direct responsibility of Subcommittee F05.03 on Research.

Current edition approved Jan. 10, 2003. Published March 2003.

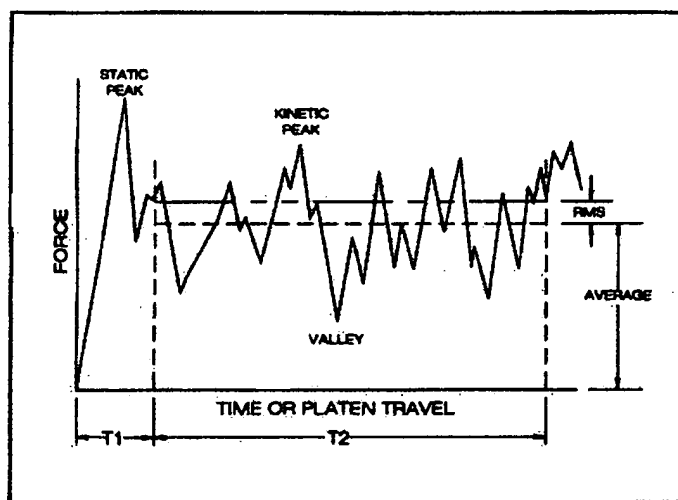


FIG. 1 Data Trace

the time required makes this method impractical for most materials. The peel strength method allows comparative studies of prints and laminating films.

4.2 Good adhesion is prime consideration for laminating films and prints. A laminating film, which does not adhere to a print or vice versa generally, has no commercial value. This method is used to obtain comparative data of peel strength of encapsulated or laminated media.

4.3 Peel strength can be measured up to a point where the peel strength is equal to the tensile strength of the adhesive or the print.

4.4 In many applications, having sufficient laminate adhesion strength is important to give satisfactory performance. The property is also important in determining the uniformity of quality.

4.5 A rough or raspy peel test will produce a higher RMS value than one that is smooth and continuous. It can be related to "zippering," "shocking," or "stick-slip."

4.6 *Bond Strength and Interfacial Adhesion*—The bond strength of a laminate essentially depends on both the adhesive and the cohesive strength of the laminating system. Peel strength of the laminate is typically determined by debonding the laminate using 180° peel test. Fig. 2 presents five possible modes of failure: (1) debonding of the ink receptive underlayer from the substrate (2) debonding of the ink receptive top layer from the underlayer, (3) debonding of the adhesive from the top ink receptive surface, (4) debonding the adhesive from the laminating film, (5) the substrate or film failure tear. The failure of the laminate will occur at its weakest link. It is generally

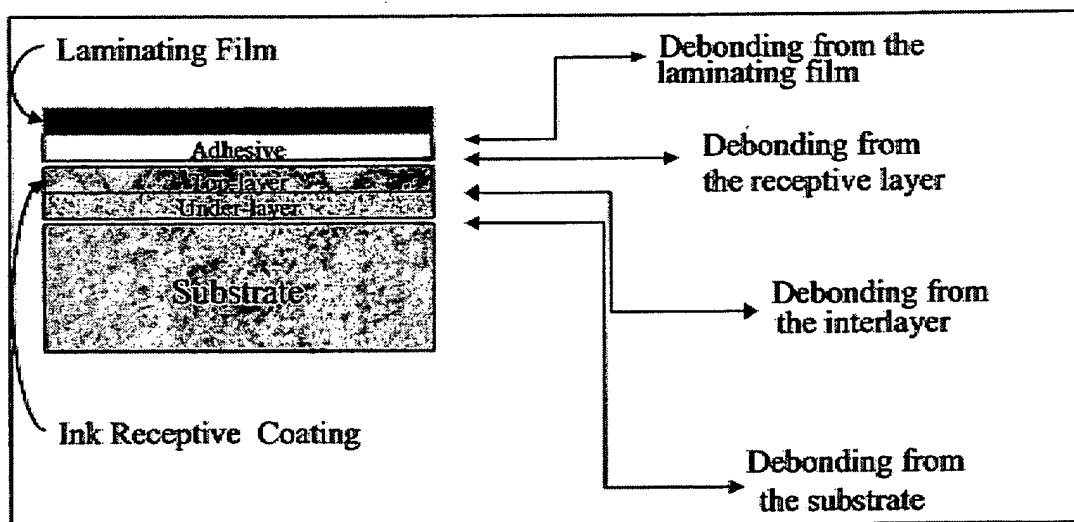


FIG. 2 Modes of Laminate Failure

desirable to have high bond strength to the recording element with failure occurring where the adhesive debonds from the ink receptive.

5. Interferences

5.1 Since the ability of laminating film to adhere to printed media is dependent on temperature and humidity, it is important that lamination be assessed under the conditions appropriate to the end use applications. While printed media may be handled and displayed under a variety of conditions, this test practice is intended to measure peel strength in typical office environments.

5.2 It is recognized that the peel strength of the laminate to the printed media is dependent on toner or ink color, toner or ink load, temperature and relative humidity. Additionally, it is dependent on the substrate, type and coat weight of absorptive layer and the colorant type (dye versus pigment). Consequently, test results must be determined individually for each printed recording media/laminate.

5.3 The peel strength of the laminate is dependent on lamination temperature and should be tested at the manufacturers recommended temperature for the best performance.

5.4 Using a laminating film thicker than 75 μ (3 mil) can create a problem in obtaining a 180° angle at the start of the test. A starting angle of less than 180° can significantly change the peel speed and mostly lead to delamination from the support instead of the coating (tearing of paper base).

5.5 The variation in recording and laminating elements requires carrying out comparative tests under well-defined conditions.

5.6 Because these measurements concern surfaces, their condition is critical to the values obtained. Be sure to keep all print surfaces clean and free from contaminants, including fingerprints.

5.7 One of the most significant (and occasionally ignored) influences on peel values is the time that elapses from lamination until the test is performed. Values can change by 100 % depending on this “aging.” Peel tests intrinsically produce quite a bit of data scatter from test to test. Keep a close watch on the results to be sure that your results are truly representative of your materials. Kinetic Peak and Valley provide important information about uniformity of lamination of the tested specimen.

5.8 Paper media have a tendency to tear the support after initial delamination from the bonding interface. It is well documented that at the same coat weight of ink receiving material, lighter paper media have higher tendency to support failure than heavier ones. Static Peak can be very useful in evaluation lamination performance of paper media. It is recommended to use Static Peak values to compare media having paper base failure. Additionally, it can also be used in evaluating media having delamination from the bonding interface. Media having paper failure but high Static Peak values are considered superior than media delaminated from the bond interface but having low peel strength.

5.9 Reducing laminating temperature could eliminate paper media failure. Lower laminating temperature reduces bonding strength between media and the laminate. In most cases, paper failure occurs when the laminate bonding strength is very high.

PRINTING

6. Test Specimen

6.1 The substrate, method of printing, ink or toner lay down, and handling of printed specimens shall be consistent with their anticipated end use.

6.2 The test image may be generated with personal computer using drawing/graphics, or page layout software able to generate composite black, saved as print file for each printer/method of printing (contributing its unique ink and ink/receiver interactions that may impact lamination.) Each print file should have its filename, type, and version identified in the image area and a place for experimental notes, for example, time, printer, environmental conditions, operator. The printer setting and a trial print of each print file version should be archived.

6.3 The recommended test image should consist of three primary and three secondary color strips (25.4 by 215.9 mm) plus a non-printed strip (25.4 by 215.9 mm). The colors should be printed in parallel in the following order: black (K), cyan, magenta, yellow, blue, green, red, white, composite black (C).

6.4 It is recommend to print in landscape mode having the 25.4-mm strips perpendicular to paper machine direction.

6.5 The test image used in comparison printers or inks (media laminated with the same film) should provide the same color elements.

7. Procedure

7.1 *Preparation of Printer*—When using an ink jet printer, print heads should be aligned, calibrated and checked for any nozzle clogging. Nozzle failure will reduce the ink lay down and can change peel strength.

7.2 The color strips printed should be generated using print files containing the appropriate printer setup specific for each application.

7.3 It is recommended that the color strip be printed as Postscript 3 file without color corrections using standard ink load limits or media selection. The printing mode is dependent on media type such as glossy photo, semi-glossy, paper bond, heavy-coated bond, and so forth.

7.4 Printing mode of the test image should be the same as recommended for media applications and available associated literature or a flyer.

7.5 Potential variables, such as temperature and relative humidity, must be monitored and controlled to guard against sample-induced changes.

7.6 Printing should be carried out at 23°C and 50 % relative humidity.

7.7 Printing can be done at extreme conditions such as 15°C, 20 % relative humidity and 38°C, 80 % relative humidity, but information about these conditions should be recorded with the lamination data.

8. Conditioning

8.1 It is recommended that samples be conditioned at 23°C and 50 % relative humidity for at least 24 h prior to printing and for at least 24 h subsequent to lamination. Specimens should be visually inspected for color uniformity and surface irregularities, which could adversely affect color densities and subsequently lamination.

8.2 The above conditioning step is pertinent only where media evaluation or comparison is needed. Obviously, as a production tool, the conditioning period is not practical. Therefore, when media are coated in production, the specimens should be obtained from each roll and checked as soon as possible in a repeatable procedure appropriate to the operation. It is imperative that operators use caution in selecting and preparing specimens to maintain good uniformity.

LAMINATION

9. Apparatus

9.1 A laminator used for continuous laminating, mounting and encapsulating. The laminator should have precise temperature control and different operating speeds to ensure that outgassing from inks does not effect adhesion or cause bubbles in the print. The laminator should:

9.2 Be capable of applying heat-activated or pressure-sensitive materials, or a combination of both.

9.3 Have an infinitely variable nip opening for all materials up to 40 mm (1.5 in.).

9.4 Have a digital or analog readout of speed.

9.5 Downward pressure and vacuum table are recommended.

10. Procedure

10.1 The print samples should be laminated 24 h after printing. The actual lamination time after printing should be presented in the report.

10.2 Before lamination, a strip of paper (50.8 by 216 mm) should be placed vertically on the top of a portion of the printed test pattern for each of the colors. About half of paper strip should cover the printed area and the other half should cover the unprinted area. This procedure allows the free end of the test specimen to be separated.

10.3 Laminating films that are 75 μ (3 mil) thick should be used for comparison purposes. Thicker films: 125, 175, and 250 μ (5, 7, and 10 mils) make it difficult to obtain the 180°C angle required in the test. At lower angles the film has a tendency to rip the support instead of delaminating from the coating. Films that are 25 or 50 μ (1 or 2 mil) thick have a tendency to elongate affecting the peel speed.

10.4 The temperature of the upper and lower rollers of laminator should be measured by infrared sensor and recorded. For precise measured and repeatability of the test, the difference between set up temperature and laminating rollers temperature should not be higher than $\pm 5^\circ\text{C}$.

10.5 Speed of lamination for paper media having base weight in the range 90 to 170 gms should be 0.9 m/min (3 ft/min). Lower or higher speed can be used depending on film thickness, paper base weight, and lamination temperature.

10.6 Lamination pressure can be expressed as a half crank or bars (psi) value if a laminator is equipped with a gage.

10.7 The print should be placed centrally in the laminator to minimize temperature variation along the rollers.

10.8 The printed samples should be conditioned 24 h before lamination at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$.

10.9 The laminations can be carried out in the office environment. However, samples printed and conditioned at

different temperatures and humidities other than the ones above should be handled in sealed plastic bags and immediately laminated. This procedure preserves moisture in the media and provides reproducible tests.

DELAMINATION

11. Sampling and Specimen Preparation

11.1 *Materials*—Razor blade, single edge or cutting board. Cellophane tape, 25 mm (1 in.) wide, #610 “Scotch” brand or equivalent.

11.2 Using the specimen cutter, cut nine 215.9 by 25.4 mm (8.5 by 1 in.) specimens for each color and unprinted background from the test image.

11.3 Prepare three test specimens for each lamination condition.

12. Conditioning

12.1 Normally, condition all laminate specimens 24 h by exposure to a relative humidity of $50 \pm 5\%$ at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) to provide additional time for equilibration of adhesive containing interfaces.

12.2 The above conditioning step is pertinent only when doing comparative testing where laminate films and media evaluation or comparison is needed. Obviously, as a production tool, the conditioning period is not practical. Therefore, when laminate adhesion is measured in production, the specimens should be obtained from each laminate roll and checked as soon as possible in a repeatable procedure appropriate to the operation.

13. Apparatus

13.1 A constant-rate-of-extension (CER) tension tester available from Instrumentors, Inc. Strongsville, OH, adequate load range to handle all materials of this type, appropriate grips, and variable or at least 304.8 mm (12 in.) per minute test speed. The tester shall have two clamps (or one) with centers in the same plane, parallel with the direction of the motion of the stressing clam, and so aligned that they will hold the specimen wholly in the same plane.

13.2 Ensure that the peel tester is level.

13.3 Follow the manufacturer’s instructions for zeroing and calibrating the peel tester and setting it to the proper speed and load range to properly measure the anticipated load.

13.4 The instrument shall be calibrated to an accuracy of 0.5 % of full scale and the readings are between 30 and 90 % of the full load range.

14. Procedure

14.1 The hardware setup for a typical 180° peel test is shown in Fig. 3.

14.2 The testing speed should be of 304.8 mm (12 in.) per minute, although higher speeds can be used for release force measurements.

14.3 Conduct testing as soon as possible after removal of the test specimens from the conditioning atmosphere and preferably under the same environmental conditions.

14.4 To maintain a separation rate of 304.8 mm (12 in.) per minute, specimens must be relatively non-extensible in the

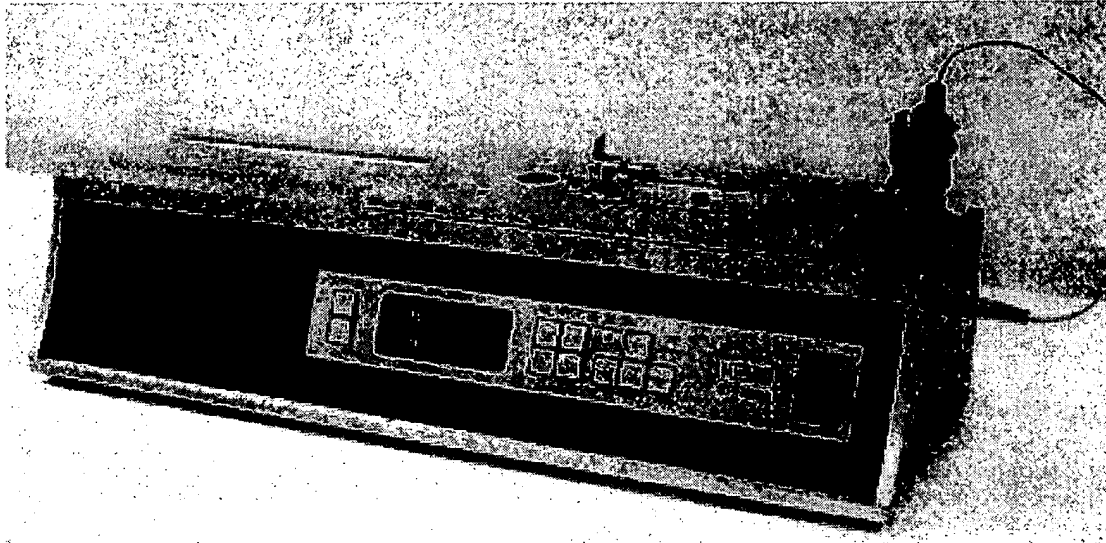


FIG. 3 Constant-Rate-of-Extension (CER) Tension Tester

expected loading range. Where a material is sufficiently extensible (that is, stretch is greater than about 15 %) to radically lower the stripping rate, reinforce the extensible member with 25.4 mm (1 in.) width non-extensible tape or change the thickness of laminating film. In reporting such a test, the backing material and method must be completely identified. It is recommended to use 3 mil laminating film for media evaluation.

14.5 Cohesive or adhesive failure may be determined by observation. Cohesive failure refers to failure in the adhesive or specimen material itself. Adhesive failure refers to the lack of adherence between materials.

14.6 Paper strips placed on top of the test image before lamination allow the free ends to be separated from the test specimen (by hand) for a distance of approximately 51 mm (2 in.). It is not recommended to initiate delamination by pulling the coating from the substrate or separating the plies of the lamination.

14.7 Place the specimen in the tester by clamping the free end of the film with the protective paper in the grip. Attach the free end of the print to the platform by pressure-sensitive tape. The peel strength of the tape has to be higher than the peel strength of the test specimen.

14.8 Align the free ends of the specimen symmetrically in the grips so that the tension is distributed uniformly.

14.9 During the actual peel test procedure, it takes a small, but finite, amount of time for the peel forces to reach a dynamic equilibrium. The selectable Delay Time for the instrument inhibits the measurement of kinetic peak, valley, average and PMS values during the Delay Time. For most standards work, a delay of 1 or 2 seconds is satisfactory. For higher speeds, a 1 second may "waste" too much of the sample.

14.10 Begin the test. After the platform starts moving, and at the end of your selected Delay Time, the Averaging light will come on, persisting for the length of time that you selected as Average Time.

14.11 At the end of that time, the platen will stop (depending on how you set up the (Platen Stop Mode) and the Results screen will display the test data.

14.12 Observe the peel force over a separation distance of at least 101.6 mm (4 in.) or the average time of at least 10 s. However, the time of 20 s is recommended. Then stop the tester and return the movable grip to its starting position. Remove the tested specimen.

14.12.1 The Average Time that you use will be dependent on the testing speed, the sample length, and, in certain instances, how much test time you can budget for each test. For lamination work, 10 s Average Time is a good value to start out with.

14.13 If the RMS is higher than 10 % of average load, the test should be repeated.

14.14 Repeat steps 14.3-14.12 with remaining specimens.

15. Interpretation of Results

15.1 Determine the peel or stripping strength for each specimen in Newtons per meter (or grams per 1 in.) width. For standard 25.4 mm width specimens, the peel value is equal to the recorded load. The average peel over the entire separation distance is the generally preferred value.

15.2 For each series of specimens, calculate the arithmetic average of all of the values obtained as the average for each color specimen and unprinted one.

15.3 Root-Mean-Square (RMS) is calculated according to the following formula:

$$RMS = \sqrt{\frac{\sum_{i=1}^N (L_N - L_{AVG})^2}{N}} \quad (1)$$

where:

- L_N = instantaneous force sample,
- L_{AVG} = previously determined mean of all N readings, and
- N = total number of data samples.

NOTE 1—The type of equipment used to determine peel strength must be stated.

16. Report

16.1 Report the following information:

16.1.1 Specimen identification, including the printer, method of printing, and the media type.

16.1.2 Lamination temperature, speed, applied pressure on nip, relative humidity and temperature in the room, where lamination is carried out, and time after printing.

16.1.3 Individual test loads.

16.1.4 Average peel or stripping strength in Newtons per meter (or grams per 1 in.) width for each color and the unprinted area.

16.1.5 Type of failure (see 4.6).

16.1.6 In the case of paper failure the value of static peak should be also reported.

16.1.7 Any unusual characteristics. Include backing if required and the conditioning cycle if other than standard.

17. Precision and Bias

17.1 A statement of bias is not applicable in view of the unavailability of a standard reference for these properties.

18. Keywords

18.1 accelerate aging; adhesion; bond strength; debonding; delamination; encapsulating; high temperature laminates; ink jet; lamination; low temperature laminates; media; mounting; peel strength; pressure-sensitive laminates; printing; thickness

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Designation: D 3359 – 02

Attachment B
USSN 10/068,824

Standard Test Methods for Measuring Adhesion by Tape Test¹

This standard is issued under the fixed designation D 3359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film.

1.2 Test Method A is primarily intended for use at job sites while Test Method B is more suitable for use in the laboratory. Also, Test Method B is not considered suitable for films thicker than 5 mils (125 μ m).

NOTE 1—Subject to agreement between the purchaser and the seller, Test Method B can be used for thicker films if wider spaced cuts are employed.

1.3 These test methods are used to establish whether the adhesion of a coating to a substrate is at a generally adequate level. They do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required.

NOTE 2—It should be recognized that differences in adherability of the coating surface can affect the results obtained with coatings having the same inherent adhesion.

1.4 In multicoat systems adhesion failure may occur between coats so that the adhesion of the coating system to the substrate is not determined.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and

Related Coating Products²

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1000 Test Method For Pressure-Sensitive Adhesive-Coated Tapes Used for Electrical and Electronic Applications³

D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴

D 2092 Guide for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁵

D 2370 Test Method for Tensile Properties of Organic Coatings²

D 3330 Test Method for Peel Adhesion of Pressure-Sensitive Tape⁶

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials²

D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser²

3. Summary of Test Methods

3.1 *Test Method A*—An X-cut is made through the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale.

3.2 *Test Method B*—A lattice pattern with either six or eleven cuts in each direction is made in the film to the substrate, pressure-sensitive tape is applied over the lattice and then removed, and adhesion is evaluated by comparison with descriptions and illustrations.

4. Significance and Use

4.1 If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because the substrate and its surface preparation (or lack of it) have a drastic effect on the adhesion of coatings, a method to evaluate adhesion of a coating to different substrates or surface treatments, or of different coatings to the

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Aug. 10, 2002. Published October 2002. Originally published as D 3359 – 74. Last previous edition D 3359 – 97.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 10.01.

⁴ *Annual Book of ASTM Standards*, Vol 02.05.

⁵ *Annual Book of ASTM Standards*, Vol 06.02.

⁶ *Annual Book of ASTM Standards*, Vol 15.09.

same substrate and treatment, is of considerable usefulness in the industry.

4.2 The limitations of all adhesion methods and the specific limitation of this test method to lower levels of adhesion (see 1.3) should be recognized before using it. The intra- and inter-laboratory precision of this test method is similar to other widely-accepted tests for coated substrates (for example, Test Method D 2370 and Test Method D 4060), but this is partly the result of it being insensitive to all but large differences in adhesion. The limited scale of 0 to 5 was selected deliberately to avoid a false impression of being sensitive.

TEST METHOD A—X-CUT TAPE TEST

5. Apparatus and Materials

5.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting devices. It is of particular importance that the cutting edges be in good condition.

5.2 *Cutting Guide*—Steel or other hard metal straightedge to ensure straight cuts.

5.3 *Tape*—25-mm (1.0-in.) wide semitransparent pressure-sensitive tape⁷ with an adhesion strength agreed upon by the supplier and the user is needed. Because of the variability in adhesion strength from batch-to-batch and with time, it is essential that tape from the same batch be used when tests are to be run in different laboratories. If this is not possible the test method should be used only for ranking a series of test coatings.

5.4 *Rubber Eraser*, on the end of a pencil.

5.5 *Illumination*—A light source is helpful in determining whether the cuts have been made through the film to the substrate.

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the adhesion is to be evaluated.

6.2 For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the adhesion.

NOTE 3—Applicable test panel description and surface preparation methods are given in Practice D 609 and Practices D 1730 and D 2092.

NOTE 4—Coatings should be applied in accordance with Practice D 823, or as agreed upon between the purchaser and the seller.

NOTE 5—If desired or specified, the coated test panels may be subjected to a preliminary exposure such as water immersion, salt spray, or high humidity before conducting the tape test. The conditions and time of exposure will be governed by ultimate coating use or shall be agreed upon between the purchaser and seller.

7. Procedure

7.1 Select an area free of blemishes and minor surface imperfections. For tests in the field, ensure that the surface is

clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

7.1.1 For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

7.2 Make two cuts in the film each about 40 mm (1.5 in.) long that intersect near their middle with a smaller angle of between 30 and 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion.

7.3 Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another X in a different location. Do not attempt to deepen a previous cut as this may affect adhesion along the incision.

7.4 Remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

7.5 Place the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of a pencil. The color under the transparent tape is a useful indication of when good contact has been made.

7.6 Within 90 ± 30 s of application, remove the tape by seizing the free end and pulling it off rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

7.7 Inspect the X-cut area for removal of coating from the substrate or previous coating and rate the adhesion in accordance with the following scale:

- 5A No peeling or removal,
- 4A Trace peeling or removal along incisions or at their intersection,
- 3A Jagged removal along incisions up to 1.6 mm (1/16 in.) on either side,
- 2A Jagged removal along most of incisions up to 3.2 mm (1/8 in.) on either side,
- 1A Removal from most of the area of the X under the tape, and
- 0A Removal beyond the area of the X.

7.8 Repeat the test in two other locations on each test panel. For large structures make sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

7.9 After making several cuts examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone before using again. Discard cutting tools that develop nicks or other defects that tear the film.

8. Report

8.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred that is, between first coat and substrate, between first and second coat, etc.

8.2 For field tests report the structure or article tested, the location and the environmental conditions at the time of testing.

8.3 For test panels report the substrate employed, the type of coating, the method of cure, and the environmental conditions at the time of testing.

8.4 If the adhesion strength of the tape has been determined in accordance with Test Methods D 1000 or D 3330, report the

⁷ Permacel 99, manufactured by Permacel, New Brunswick, NJ 08903, and available from various Permacel tape distributors, is reported to be suitable for this purpose. The manufacturer of this tape and the manufacturer of the tape used in the interlaboratory study (see RR: D01-1008), have advised this subcommittee that the properties of these tapes were changed. Users of it should, therefore, check whether current material gives comparable results to previous supplied material.

results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

8.5 If the test is performed after immersion, report immersion conditions and method of sample preparation.

9. Precision and Bias ⁸

9.1 In an interlaboratory study of this test method in which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion, the within-laboratories standard deviation was found to be 0.33 and the between-laboratories 0.44. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than 1 rating unit for two measurements.

9.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 1.5 rating units.

9.2 Bias cannot be established for these test methods.

TEST METHOD B—CROSS-CUT TAPE TEST

10. Apparatus and Materials

10.1 *Cutting Tool*⁹—Sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between 15 and 30° that will make either a single cut or several cuts at once. It is of particular importance that the cutting edge or edges be in good condition.

10.2 *Cutting Guide*—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straight-edge or template to ensure straight cuts.

10.3 *Rule*—Tempered steel rule graduated in 0.5 mm for measuring individual cuts.

10.4 *Tape*, as described in 5.3.

10.5 *Rubber Eraser*, on the end of a pencil.

10.6 *Illumination*, as described in 5.5.

10.7 *Magnifying Glass*—An illuminated magnifier to be used while making individual cuts and examining the test area.

11. Test Specimens

11.1 Test specimens shall be as described in Section 6. It should be noted, however, that multitip cutters¹⁰ provide good results only on test areas sufficiently plane that all cutting edges contact the substrate to the same degree. Check for flatness with a straight edge such as that of the tempered steel rule (10.3).

⁸ Supporting data are available from ASTM International Headquarters. Request RR: D01-1008.

⁹ Multiblade cutters are available from a few sources that specialize in testing equipment for the paint industry. One supplier that has assisted in the refinement of these methods is given in footnote 10.

¹⁰ The sole source of supply of the multitip cutter for coated pipe surfaces known to the committee at this time is Paul N. Gardner Co., 316 NE First St., Pompano Beach, FL 33060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

12. Procedure

12.1 Where required or when agreed upon, subject the specimens to a preliminary test before conducting the tape test (see Note 3). After drying or testing the coating, conduct the tape test at room temperature as defined in Specification D 3924, unless D 3924 standard temperature is required or agreed.

12.1.1 For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

12.2 Select an area free of blemishes and minor surface imperfections, place on a firm base, and under the illuminated magnifier, make parallel cuts as follows:

12.2.1 For coatings having a dry film thickness up to and including 2.0 mils (50 μ m) space the cuts 1 mm apart and make eleven cuts unless otherwise agreed upon.

12.2.2 For coatings having a dry film thickness between 2.0 mils (50 μ m) and 5 mils (125 μ m), space the cuts 2 mm apart and make six cuts. For films thicker than 5 mils use Test Method A.¹¹

12.2.3 Make all cuts about 20 mm ($\frac{3}{4}$ in.) long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area.

12.3 After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings.

12.4 Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts.

12.5 Brush the area as before and inspect the incisions for reflection of light from the substrate. If the metal has not been reached make another grid in a different location.

12.6 Remove two complete laps of tape and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

12.7 Place the center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser on the end of a pencil. The color under the tape is a useful indication of when good contact has been made.

12.8 Within 90 ± 30 s of application, remove the tape by seizing the free end and rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

12.9 Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. Rate the adhesion in accordance with the following scale illustrated in Fig. 1:

¹¹ Test Method B has been used successfully by some people on coatings greater than 5 mils (0.13 mm) by spacing the cuts 5 mm apart. However, the precision values given in 14.1 do not apply as they are based on coatings less than 5 mm (0.13 mm) in thickness.

- 5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
- 4B Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
- 3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
- 2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
- 1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
- 0B Flaking and detachment worse than Grade 1.

12.10 Repeat the test in two other locations on each test panel.

13. Report

13.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred, that is, between first coat and substrate, between first and second coat, etc.

13.2 Report the substrate employed, the type of coating and the method of cure.

13.3 If the adhesion strength has been determined in accordance with Test Methods D 1000 or D 3330, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

13.4 If the test is performed after immersion, report immersion conditions and method of sample preparation.

14. Precision and Bias ⁸

14.1 On the basis of two interlaboratory tests of this test method in one of which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion and in the other operators in six laboratories made three measurements on two panels each of four different coatings applied over two other coatings, the pooled standard deviations for within- and between-laboratories were found to be 0.37 and 0.7. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than one rating unit for two measurements.

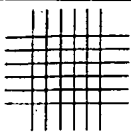
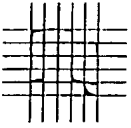
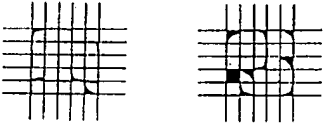
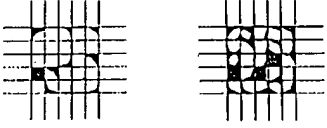
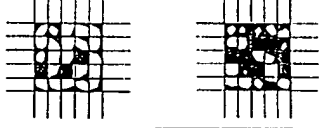
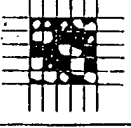
CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

14.1.2 *Reproducibility*—Two results, each the mean of duplicates or triplicates, obtained by different operators should be considered suspect if they differ by more than two rating units.

14.2 Bias cannot be established for these test methods.

15. Keywords

15.1 adhesion; crosscut adhesion test method; tape; tape adhesion test method; X-cut adhesion test method

APPENDIX

(Nonmandatory Information)

X1. COMMENTARY

X1.1 Introduction

X1.1.1 Given the complexities of the adhesion process, can adhesion be measured? As Mittal (1)¹² has pointed out, the answer is both yes and no. It is reasonable to state that at the present time no test exists that can precisely assess the actual physical strength of an adhesive bond. But it can also be said that it is possible to obtain an indication of relative adhesion performance.

X1.1.2 Practical adhesion test methods are generally of two types: “*implied*” and “*direct*.” “Implied” tests include indentation or scribe techniques, rub testing, and wear testing. Criticism of these tests arises when they are used to quantify the strength of adhesive bonding. But this, in fact, is not their purpose. An “implied” test should be used to assess coating performance under actual service conditions. “Direct” measurements, on the other hand, are intended expressly to measure adhesion. Meaningful tests of this type are highly sought after, primarily because the results are expressed by a single discrete quantity, the force required to rupture the coating/substrate bond under prescribed conditions. Direct tests include the Hesiometer and the Adherometer (2). Common methods which approach the direct tests are peel, lap-shear, and tensile tests.

X1.2 Test Methods

X1.2.1 In practice, numerous types of tests have been used to attempt to evaluate adhesion by inducing bond rupture by different modes. Criteria deemed essential for a test to warrant large-scale acceptance are: use of a straightforward and unambiguous procedure; relevance to its intended application; repeatability and reproducibility; and quantifiability, including a meaningful rating scale for assessing performance.

X1.2.2 Test methods used for coatings on metals are: peel adhesion or “tape testing;” Gardner impact flexibility testing; and adhesive joint testing including shear (lap joint) and direct tensile (butt joint) testing. These tests do not strictly meet all the criteria listed, but an appealing aspect of these tests is that in most cases the equipment/instrumentation is readily available or can be obtained at reasonable cost.

X1.2.3 A wide diversity of tests methods have been developed over the years that measure aspects of adhesion (1-5). There generally is difficulty, however, in relating these tests to basic adhesion phenomena.

X1.3 The Tape Test

X1.3.1 By far the most prevalent test for evaluating coating “adhesion” is the tape-and-peel test, which has been used since the 1930’s. In its simplest version a piece of adhesive tape is pressed against the paint film and the resistance to and degree

of film removal observed when the tape is pulled off. Since an intact film with appreciable adhesion is frequently not removed at all, the severity of the test is usually enhanced by cutting into the film a figure X or a cross hatched pattern, before applying and removing the tape. Adhesion is then rated by comparing film removed against an established rating scale. If an intact film is peeled cleanly by the tape, or if it debonds just by cutting into it without applying tape, then the adhesion is rated simply as poor or very poor, a more precise evaluation of such films not being within the capability of this test.

X1.3.2 The current widely-used version was first published in 1974; two test methods are covered in this standard. Both test methods are used to establish whether the adhesion of a coating to a substrate is at an adequate level; however they do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required. Major limitations of the tape test are its low sensitivity, applicability only to coatings of relatively low bond strengths, and non-determination of adhesion to the substrate where failure occurs within a single coat, as when testing primers alone, or within or between coats in multicoat systems. For multicoat systems where adhesion failure may occur between or within coats, the adhesion of the coating system to the substrate is not determined.

X1.3.3 Repeatability within one rating unit is generally observed for coatings on metals for both methods, with reproducibility of one to two units. The tape test enjoys widespread popularity and is viewed as “simple” as well as low in cost. Applied to metals, it is economical to perform, lends itself to job site application, and most importantly, after decades of use, people feel comfortable with it.

X1.3.4 When a flexible adhesive tape is applied to a coated rigid substrate surface and then removed, the removal process has been described in terms of the “peel phenomenon,” as illustrated in Fig. X1.1.

X1.3.5 Peeling begins at the “toothed” leading edge (at the right) and proceeds along the coating adhesive/interface or the coating/substrate interface, depending on the relative bond strengths. It is assumed that coating removal occurs when the tensile force generated along the latter interface, which is a function of the rheological properties of the backing and adhesive layer materials, is greater than the bond strength at the coating-substrate interface (or cohesive strength of the coating). In actuality, however, this force is distributed over a discrete distance (O-A) in Fig. X1.1, which relates directly to the properties described, not concentrated at a point (O) in Fig. X1.1 as in the theoretical case—though the tensile force is greatest at the origin for both. A significant compressive force arises from the response of the tape backing material to being stretched. Thus both tensile and compressive forces are involved in adhesion tape testing.

X1.3.6 Close scrutiny of the tape test with respect to the

¹² The boldface numbers in parentheses refer to the list of references at the end of this test method.

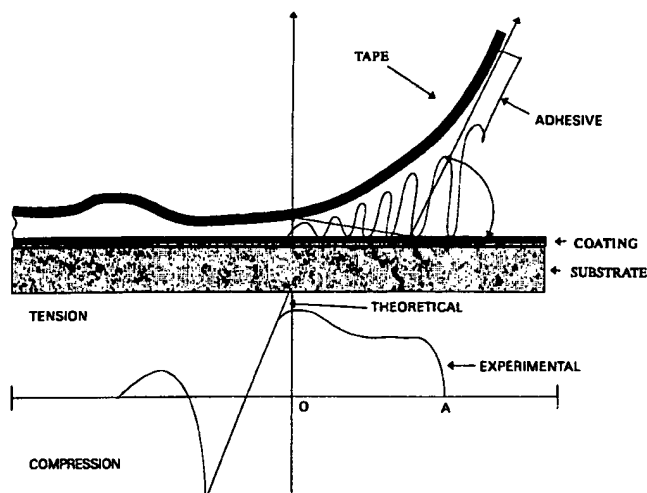


FIG. X1.1 Peel Profile (6)

nature of the tape employed and certain aspects of the procedure itself reveal several factors, each or any combination of which can dramatically affect the results of the test as discussed (6).

X1.4 Peel Adhesion Testing on Plastic Substrates

X1.4.1 Tape tests have been criticized when used for substrates other than metal, such as plastics. The central issues are that the test on plastics lacks reproducibility and does not relate to the intended application. Both concerns are well founded: poor precision is a direct result of several factors intrinsic to the materials employed and the procedure itself. More importantly, in this instance the test is being applied beyond its intended scope. These test methods were designed for relatively ductile coatings applied to metal substrates, not for coatings (often brittle) applied to plastic parts (7). The unique functional requirements of coatings on plastic substrates cause the usual tape tests to be unsatisfactory for measuring adhesion performance in practice.

X1.5 The Tape Controversy

X1.5.1 With the withdrawal from commerce of the tape specified originally, 3M No. 710, current test methods no longer identify a specific tape. Differences in tapes used can lead to different results as small changes in backing stiffness and adhesive rheology cause large changes in the tension area. Some commercial tapes are manufactured to meet minimum standards. A given lot may surpass these standards and thus be suitable for general market distribution; however, such a lot may be a source of serious and unexpected error in assessing adhesion. One commercially available tape test kit had included a tape with adhesion strength variations of up to 50 % claimed by the manufacturer. Also, because tapes change on storage, bond strengths of the tape may change over time (7, 8).

X1.5.2 While there are tapes available that appear to deliver consistent performance, a given tape does not adhere equally well to all coatings. For example, when the peel removal force of the tape (from the coating) used earlier by Task Group D01.23.10 to establish precision of the method, by 3M No. 710 was examined with seven different electromagnetic

interference/radio frequency interference (EMI/RFI) coatings, it was found that, while peel was indeed consistent for a given coating, the value varied by 25 % between the highest and lowest ratings among coatings. Several factors that contribute to these differences include coating composition and topology: as a result, no single tape is likely to be suitable for testing all coatings. Further, the tape test does not give an absolute value for the force required for bond rupture, but serves only as an indicator that some minimum value for bond strength was met or exceeded (7, 8).

X1.6 Procedural Problems

X1.6.1 The tape test is operator intensive. By design it was made as simple as possible to perform, and requires a minimum of specialized equipment and materials that must meet certain specifications. The accuracy and precision depend largely upon the skill of the operator and the operator's ability to perform the test in a consistent manner. Key steps that directly reflect the importance of operator skill include the angle and rate of tape removal and the visual assessment of the tested sample. It is not unexpected that different operators might obtain different results (7, 8).

X1.6.2 Peel Angle and Rate:

The standard requires that the free end of the tape be removed rapidly at as close to a 180° angle as possible. If the peel angle and rate vary, the force required to remove the tape can change dramatically. Nearly linear increases were observed in peel force approaching 100 % as peel angle was changed from 135 to 180, and similar large differences can be expected in peel force as peel rate varies. These effects are related as they reflect certain rheological properties of the backing and adhesive that are molecular in origin. Variation in pull rate and peel angle can effect large differences in test values and must be minimized to assure reproducibility (9).

X1.6.3 Visual Assessment:

The final step in the test is visual assessment of the coating removed from the specimen, which is subjective in nature, so that the coatings can vary among individuals evaluating the same specimen (9).

X1.6.3.1 Performance in the tape test is based on the amount of coating removed compared to a descriptive scale. The exposure of the substrate can be due to factors other than coating adhesion, including that arising from the requirement that the coating be cut (hence the synonym "cross-hatch adhesion test"). Justification for the cutting step is reasonable as cutting provides a free edge from which peeling can begin without having to overcome the cohesive strength of the coating layer.

X1.6.3.2 Cutting might be suitable for coatings applied to metal substrates, but for coatings applied to plastics or wood, the process can lead to a misleading indication of poor adhesion due to the unique interfacial zone. For coatings on soft substrates, issues include how deep should this cut penetrate, and is it possible to cut only to the interface?

X1.6.3.3 In general, if adhesion test panels are examined microscopically, it is often clearly evident that the coating removal results from substrate failure at or below the interface, and not from the adhesive failure between the coating and the substrate. Cohesive failure within the coating film is also

frequently observed. However, with the tape test, failures within the substrate or coating layers are rare because the tape adhesive is not usually strong enough to exceed the cohesive strengths of normal substrates and organic coatings. Although some rather brittle coatings may exhibit cohesive failure, the tape test adhesion method does not make provision for giving failure locality (7, 8).

X1.6.4 Use of the test method in the field can lead to

variation in test results due to temperature and humidity changes and their effect upon tape, coating and substrate.

X1.7 Conclusion

X1.7.1 All the issues aside, if these test methods are used within the Scope Section and are performed carefully, some insight into the approximate, relative level of adhesion can be gained.

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SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D 3359 - 97) that may impact the use of this standard.

- | | |
|--|--|
| <p>(1) Deleted reference to Test Method D 2197 in Referenced Documents section and editorially changed footnote 10 to avoid confusion with another adhesion test method.</p> | <p>(2) Added 7.1.1, 8.5, 12.1.1, and 13.4 to clarify use when testing samples that have been immersed.</p> |
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Attachment C-2
USSN 10/068,824

SC5-0947

SC-5 COATING REQUEST

Coating Sheet 1

Originator
C Romano/ K Maskasky

Support: RC Paper

Conditions	DB	DP	Pres	Ft
Setting Sect.	40	26	0.3	39
Dryer No. 1	120	40	1	32
Dryer No. 2	120	50	2	38

UserID	Phone	Pager	Prob.#
L125610 / L594389	x23004 / x77880	x50938	0700-03058

Date: 8/8/01
Coated by: *Martina*

Part ID	SLOT Melt #	Slide Melt #	Melt Vol.	% solids	ml/ft2	Pump RPM	Ctg. spd	Melt Experiment	Laydown (mg/ft2)	Linear Feet	Notes
1	A	1	300	2.5%	4.0	53.8	12	Z-210/POL-4455	100	15	Coat over gel rug
2	A	2	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
3	A	3	300	2.5%	4.0	53.8	12	Z-210/BAeMn	100	15	
4	A	4	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
5	A	5	300	2.5%	4.0	53.8	12	Z-210/AeMn	100	15	
6	A	6	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
7	A	7	300	2.5%	4.0	53.8	12	Z-210/Glascal C44	100	15	
8	A	8	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
9	A	9	300	2.5%	4.0	53.8	12	Z-210/Glascal RP3	100	15	
10	A	10	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
11	A	11	300	2.5%	4.0	53.8	12	Elvanol 52-22/Witcobond 232	100	15	Example 4
12	A	12	300	2.5%	4.0	40.4	12	Z-210/Lucidene 243	100	15	
13	A	13	300	2.5%	4.0	53.8	12	Elvanol 52-22/Witcobond 232	150	15	
14	A	14	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	100	15	
15	A	15	300	2.5%	4.0	53.8	12	Z-210/Morceryl 132	100	15	
16	A	16	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
17	A	17	300	2.5%	4.0	53.8	12	Z-210/U710	100	15	
18	A	18	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
19	A	19	300	2.5%	4.0	53.8	12	Z-210/U410	100	15	
20	A	20	300	2.5%	4.0	40.4	12	Elvanol 52-22/Witcobond 232	150	15	
21	A	21	300	2.5%	4.0	53.8	12	Z-210/AC-2538	100	15	

C Romano

Shirley A. Sadler 11-21-01

	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
12	12	800	2.5%	4.0	53.8	12	Z-210/UCX 01-011	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
13	13	800	2.5%	4.0	53.8	12	Z-210/UCX 99-027	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
14	14	800	2.5%	4.0	53.8	12	Z-210/Witcobond 253	100	Example 3
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
15	15	800	2.5%	4.0	53.8	12	Z-210/WD30	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
16	16	800	2.5%	4.0	53.8	12	Z-210/DP6-7133	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
17	17	800	2.5%	4.0	53.8	12	DP6-7133	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
18	18	800	2.5%	4.0	53.8	12	Z-210	100	Example 2
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
*19	19	800	2.5%	4.0	53.8	12	Z-200	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
20	20	800	2.5%	4.0	53.8	12	Z-320	100	Example 1
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
21	21	800	2.5%	4.0	53.8	12	OKS-3431	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
22	22	800	2.5%	4.0	53.8	12	OKS-3432	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
23	GOC	800	2.5%	4.0	53.8	12	Galaxy OC	100	15
	A	800	5%	3.0	40.4	12	Elvanol 52-22/Witcobond 232	150	
24									15
	1	800	2.5%	4.0	53.8	12	Z-210/POL-4455	100	
25									15
	1	800	2.5%	10.0	134.6	12	Z-210/POL-4455	250	

CE Romanoff

Shiela A. Sadler 11-21-01

Attachment C-3
USSN 10/068,824

SC5-0927				SC-5 COATING REQUEST			
Coating Sheet 1				Originator	C Romano/ K Maskasky	Support:	RC paper
Conditions		DB	DP	Pres	Ft	UserID	L125610 / L594389
Setting Sect.		40	26	0.3	39	Phone	x23004 / x77880
Dryer No. 1		120	40	1	32		
Dryer No. 2		120	50	2	38	Prob.# 0700-03058	
				Coated by:	Date: 26-Jul-01		
				Page Witnessed by			

Part ID	SLOT Melt #	Slide Melt #	Melt Vol.	% solids	ml/ft2	Pump RPM	Ctg. spd	Melt Experiment	Laydown (mg/ft2)	Linear Feet	Notes
1	1	OC	100	2%	5.0	67.3	12	Galaxy OC	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
2	2	OC	100	2%	5.0	67.3	12	Alcotex 864	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
3	3	OC	100	2%	5.0	67.3	12	QP300	100	12	Control Example 1
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
4	4	OC	100	2%	5.0	67.3	12	K100	100	12	Control Example 2
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
5	5	OC	100	2%	5.0	67.3	12	A15	100	12	Control Example 3
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
6	6	OC	100	2%	5.0	67.3	12	F4M	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
7	7	OC	100	2%	5.0	67.3	12	CMC	100	12	Control Example 4
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
8	8	OC	100	2%	5.0	67.3	12	Airvam	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
9	9	OC	100	2%	5.0	67.3	12	K35LV	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
10	10	OC	100	2%	5.0	67.3	12	Aquazol 5	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
11	11	OC	100	2%	5.0	67.3	12	Aquazol 50	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
12	12	IL	100	2%	5.0	67.3	12	Aquazol 200	100	12	
	A	BL	800	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12	
13	13	IL	100	2%	5.0	67.3	12	Aquazol 500	100	12	

Toxic ?: Yes	Special Instructions:										Jacketed hopper @104°F
	Melts @104°F					Melt					
	A	BL	800	2%	5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
14	14	IL	100	2%	5.0		67.3	12	AH-17	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
15	15	IL	100	2%	5.0		67.3	12	AH-26	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
16	16	IL	100	2%	5.0		67.3	12	GH-23	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
17	17	IL	100	2%	5.0		67.3	12	GL-05	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
18	18	IL	100	2%	5.0		67.3	12	K-210	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
19	19	IL	100	2%	5.0		67.3	12	KH-17	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
20	20	IL	100	2%	5.0		67.3	12	KH-20	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
21	21	IL	100	2%	5.0		67.3	12	KP-06	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
22	22	IL	100	2%	5.0		67.3	12	KP-08	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
23	23	IL	100	2%	5.0		67.3	12	Z320	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
24	24	IL	100	2%	5.0		67.3	12	N-300	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
25	25	IL	100	2%	5.0		67.3	12	T-215	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
26	26	IL	100	2%	5.0		67.3	12	W/O320	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
27	27	IL	100	2%	5.0		67.3	12	Z-100	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
28	28	IL	100	2%	5.0		67.3	12	Z-210	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12
29	29	IL	100	2%	5.0		67.3	12	Alcotex 552P	100	12
	A	BL	800		5%	3.0	40.4	12	Elvanol 52-22/Wcb 232 (77/23)	150	12

No	X	Stir	yes	Suction ??	Handroll:
----	---	------	-----	------------	-----------

+++++

(02/14/05)

[illegible]

The calculated dry weight of the batch is:

1.005

2.00%

0.100 grams per ft²40.00 ft²

50.00

190.64

1 Add 50g for each hopper

Charles E. Romano Jr 2-17-05
Karen E. Maskeby 2-17-05

```

+++++
(02/14/05)
Solution OC-02 Z-210 PVA + Wcb-244
+++++
Total Weight Solids % Item Chemical Identification Wet Weight Dry Weight % of dry Total Weight mg/ft2 Dry mg/ft2 Total mg/ft2
29.12 10.00 -1- Z-210 Acac-PVA 26.21 2.912 2.912 72.68% 29.12 654.14 72.68 726.83
2.44 39.70 -1- Wcb-244 1.47 0.970 0.970 24.22% 2.44 36.78 24.22 61.00
67.19 0.00 -2- Water 167.19 0.000 0.000 0.00% 167.19 4172.86 0.00 4172.86
0.96 10.00 -3- APG 325N 0.86 0.096 0.096 2.40% 0.96 21.59 2.40 23.99
0.28 10.00 -3- Olin 10G 0.25 0.028 0.028 0.70% 0.28 6.32 0.70 7.03
0.28
200.00
=====
The calculated dry weight of the batch is: 4.01 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 40.07 ft²
COATED FOOTAGE: 50.00
TOTAL SOLUTION NEEDED: 190.39
Add 50g for each hopper
+++++

```

Example 2

parts	ft	increase
1	40	1.25

Chad E Romano Jr 2-17-05
L.A. S.M. 2-17-05

```

+++++ (02/14/05) +++++
Solution OC-03 Z-210 PVA + Wcb-253
+++++
Total Weight Solids % Item Chemical Identification Wet Weight Dry Weight % of dry Total Weight mg/ft2 Dry mg/ft2 Total mg/ft2
29.06 10.00 -1- Z-210 Acac-PVA 26.16 2.906 72.67% 29.06 654.05 72.67 726.72
2.94 33.00 -1- Wcb-253 1.97 0.969 24.23% 2.94 49.19 24.23 73.42
66.76 0.00 -2- Water 166.76 0.000 0.00% 166.76 4169.72 0.00 4169.72
0.96 10.00 -3- APG 325N 0.86 0.096 2.40% 0.96 21.61 2.40 24.01
0.28 10.00 -3- Olin 10G 0.25 0.028 0.70% 0.28 6.30 0.70 7.00
0.28 -----
200.00 =====
=====
+++++ The calculated dry weight of the batch is: 4.00 g

```

Example 3

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 2.00%

ENTER THE DRY COVERAGE: 0.100 grams per ft2

The calculated yield of this batch is: 39.99 ft²

COATED FOOTAGE: 50.00

TOTAL SOLUTION NEEDED: 190.65

parts	ft	increase
1	40	1.25

1 Add 50g for each hopper

Charles Romano for 2-17-05
Karen S Maskester 2-17-05

+

Support Type:

4.50 inches

5.00 inches

(02/14/05)

Solution **OC-04** Z-210 PVA + Morcryn 132

Total Weight	% Solids	Item #	Chemical Identification	Wet Weight	Dry Weight	% of dry	Total Weight	Wet mg/ft2	Dry mg/ft2	Total mg/ft2
29.06	10.00	-1-	Z-210 Acac-PVA	26.16	2.906	72.68%	29.06	654.14	72.68	726.82
3.23	30.00	-1-	Morcryl 132	2.26	0.968	24.21%	3.23	56.50	24.21	80.71
166.47	0.00	-2-	Water	166.47	0.000	0.00%	166.47	4162.95	0.00	4162.95
0.96	10.00	-3-	APG 325N	0.87	0.096	2.40%	0.96	21.64	2.40	24.04
0.28	10.00	-3-	Olin 10G	0.25	0.028	0.70%	0.28	6.31	0.70	7.01
200.00				196.00	4.00	100.0%	200.00	4901.53	100.00	5001.53

The calculated dry weight of the batch is: 4.00 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 2.00%

ENTER THE DRY COVERAGE: 0.100 grams per ft2

The calculated yield of this batch is: 39.99 ft²

COATED FOOTAGE: 50.00

parts	ft	increase
1	40	1.25

TOTAL SOLUTION NEEDED:	
190.67	1. Add 50g for each hopper

Add 50g for each hopper

OC-04 Z-210 PVA +Mocryl 132.xls
2/15/20053:20 PM

Charles E Romano Jr 2-17-05
 K.A. S.M. 6-6 2-17-05

Kar. S M. L. b 7-17-75

Example 4

```

+++++ (02/14/05) +++++
Solution OC-05 HEC QP-300
+++++
Total Weight 136.65 2.84 -1- HEC QP-300 132.77 3.881 Dry Weight 136.65 100.0% 96.90% 3315.01 96.90 3411.91
62.11 0.00 -2- Water 62.11 0.000 0.00% 1550.87 0.00 1550.87
0.96 10.00 -3- APG 325N 0.87 0.096 2.40% 21.61 2.40 24.01
0.28 10.00 -3- Olin 10G 0.25 0.028 0.70% 6.30 0.70 7.00
0.28 -----
200.00 -----
=====
+++++ The calculated dry weight of the batch is: 4.00 g 1.005 2.00%
ENTER SPECIFIC GRAVITY OF THE SOLUTION->
The calculated solids of this batch is:
ENTER THE DRY COVERAGE:
The calculated yield of this batch is:
COATED FOOTAGE:
TOTAL SOLUTION NEEDED:
+++++ Add 50g for each hopper +++++

```

Control Example 1

parts	ft	increase
1	40	1.25

Charles E Romano Jr 2-17-05
H. E. W. I.

Control Example 2

Charles E Pomaro Jr 2-17-05
Karen E Maskashu 2-17-05

[illegible]

Support Type:

4.50 inches

(02/14/05)

5.00 inches

Solution OC-07 A15LV

[illegible]

The calculated dry weight of the batch is: 4.01 'a

ENTER SPECIFIC GRAVITY OF THE SOLUTION->

The calculated solids of this batch is:

ENTER THE DRY COVERAGE.

0 100 grams per ft²

The calculated yield of this batch is:

40 06 f+2

COATED FOOTAGE:

5000

parts	ft	increase
1	40	1.25

TOTAL SOLUTION NEEDED.

190 41

Add 50¢ for each honoree

Control Example 3

Charles E Romano Jr 2-17-05
Karen E Miskask 2-17-05

```

+++++++
(02/14/05)
Solution OC-08 Carbose LT-30
+++++++
Total Weight 12.44 31.20 12.44 31.20
+++++++
Wet Weight 186.33 0.00 186.33 0.00
+++++++
Chemical Identification # -1- Carbose LT-30
+++++++
Wet Weight 8.56 3.880 8.56 3.880
+++++++
Dry Weight 0.000 0.000 0.000 0.000
+++++++
% of dry 0.00% 0.00% 0.00% 0.00%
+++++++
Total Weight 186.33 0.00 186.33 0.00
+++++++
Wet Weight 21.57 2.40 21.57 2.40
+++++++
Dry Weight 6.29 0.70 6.29 0.70
+++++++
Total mg/ft2 4653.86 0.00 4653.86 0.00
+++++++
Total mg/ft2 23.97 6.99 23.97 6.99
+++++++
Support Type:
Coated Width: 4.50 inches
Support Width: 5.00 inches
+++++++

```

Control Example 4

The calculated dry weight of the batch is: 4.00 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 2.00%

ENTER THE DRY COVERAGE: 0.100 grams per ft2

The calculated yield of this batch is: 40.04 ft²

COATED FOOTAGE: 50.00

TOTAL SOLUTION NEEDED: 190.50

1 Add 50g for each hopper

parts	ft	increase
1	40	1.25

Charles E. Romano Jr 2-17-05
Karen E. Maskelesky 2-17-05

```

+++++ (02/14/05) +++++
Solution OC-09 GH-23 PVA
+++++
Total Weight Solids % Item Chemical # Identification Wet Weight Dry Weight % of dry Total Weight Wet mg/ft2 Dry mg/ft2 Total mg/ft2
+++++
38.76 10.00 -1- GH-23 PVA 34.88 3.876 96.90% 38.76 872.10 96.90 969.00
60.00 0.00 -2- Water 160.00 0.000 0.00% 160.00 4000.00 0.00 4000.00
0.96 10.00 -3- APG 325N 0.86 0.096 2.40% 0.96 21.60 2.40 24.00
0.28 10.00 -3- Olin 10G 0.25 0.028 0.70% 0.28 6.30 0.70 7.00
200.00
=====
196.00 4.00 100.0% 200.00 4900.00 100.00 5000.00
=====
+++++
The calculated dry weight of the batch is: 4.00 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 40.00 ft²
COATED FOOTAGE: 50.00
TOTAL SOLUTION NEEDED: 190.63
Add 50g for each hopper
+++++

```

Control Example 5

parts	ft	increase
1	40	1.25

Charles E Romano b 2-17-05
Karen E Maskensky 2-17-05

```

+++++ (02/14/05) ++++++
Solution OC-10 WO-320 PVA
+++++
Total Weight 59.59 6.50 -1- WO-320 PVA 55.71 3.873 96.90% 59.59 1393.85 96.90 1490.75
139.17 0.00 -2- Water 139.17 0.000 0.00% 139.17 3481.74 0.00 3481.74
0.96 10.00 -3- APG 325N 0.86 0.096 2.40% 0.96 21.61 2.40 24.01
0.28 10.00 -3- Olin 10G 0.25 0.028 0.70% 0.28 6.30 0.70 7.00
0.28 200.00 -----
=====
The calculated dry weight of the batch is: 4.00 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 39.97 ft²
COATED FOOTAGE: 50.00
TOTAL SOLUTION NEEDED: 190.72
Add 50g for each hopper

```

Control Example 6

parts	ft	increase
1	40	1.25

Charles E Fornaro 2-17-05
Karen E Monahan

```

+++++ (02/14/05) +++++
Solution OC-11 Z-320 Acetoacetylated PVA
+++++
Total Weight % Solids 30.01 10.00 119.99 0.00 0.00 10.00 0.00 10.00
+++++
Total Weight 30.01 119.99 0.00 0.00 0.00 10.00 0.00 10.00
+++++
Chemical Identification # Item
+++++
Z-320 Acac-PVA -1- 3.001 27.01 119.99 0.00 0.00 0.00 0.00
Water -2- 0.000 0.00 0.00 0.00 0.00 0.00 0.00 0.00
APG 325N -3- 0.000 0.00 0.00 0.00 0.00 0.00 0.00 0.00
Olin 10G -3- 0.000 0.00 0.00 0.00 0.00 0.00 0.00 0.00
+++++
Dry Weight 3.00 27.01 119.99 0.00 0.00 0.00 0.00 0.00
+++++
% of dry 100.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00%
+++++
Total Weight 30.01 119.99 0.00 0.00 0.00 0.00 0.00 0.00
+++++
Wet mg/ft2 900.00 3998.97 0.00 0.00 0.00 0.00 0.00 0.00
+++++
Dry mg/ft2 100.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
+++++
Total mg/ft2 1000.00 3998.97 0.00 0.00 0.00 0.00 0.00 0.00
+++++
Support Type: 4.50 inches
Coated Width: 5.00 inches
Support Width:
+++++
The calculated dry weight of the batch is: 3.00 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 30.01 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.45
Add 50g for each hopper

```

parts	ft	increase
1	30	1.25

Example 5

Charles E. Romano & 2-17-05
Karen E. W. asksky 2-17-05

```

+++++ (02/14/05) +++++
Solution OC-12 Z-210 PVA + Wcb-244
+++++
Total Weight 22.45 1.89 125.67 0.00 0.00
+++++
Item # Identification Wet Weight Dry Weight % of dry Total Weight Total mg/ft2
+++++
-1- Z-210 Acac-PVA 20.20 2.245 75.00% 22.45 674.96 75.00 749.95
-1- Wcb-244 1.14 0.748 25.00% 1.89 37.98 25.00 62.98
-2- Water 125.67 0.000 0.00% 125.67 4198.13 0.00 4198.13
-3- APG 325N 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
-3- Olin 10G 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
+++++
150.00
=====
The calculated dry weight of the batch is: 2.99 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 29.93 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.70
Add 50g for each hopper

```

parts	ft	increase
1	30	1.25

Example 6

Charles E. Romano Jr 2-17-05
 Karen E. Maskevsky 2-17-05

OC-13 Z-210 PVA + Wcb-253.xls
2/15/2005 3:22 PM

```

+++++ (02/14/05) +++++
Solution OC-14 Z-210 PVA + Morcyl 132
+++++
Total Weight Solids % Item Chemical Identification Wet Weight Dry Weight % of dry Total Weight Wet mg/ft2 Dry mg/ft2 Total mg/ft2
+++++
22.54 10.00 -1- Z-210 Acac-PVA 20.29 2.254 75.00% 22.54 675.00 75.00 750.00
2.50 30.00 -1- Morcyl 132 1.75 0.751 25.00% 2.50 58.33 25.00 83.33
124.96 0.00 -2- Water 124.96 0.000 0.00% 124.96 4157.89 0.00 4157.89
0.00 10.00 -3- APG 325N 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
0.00 10.00 -3- Olin 10G 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
=====
150.00
=====
The calculated dry weight of the batch is: 3.01 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 2.00%

ENTER THE DRY COVERAGE: 0.100 grams per ft2

The calculated yield of this batch is: 30.05 ft²

COATED FOOTAGE: 37.50

TOTAL SOLUTION NEEDED: 155.28
Add 50g for each hopper
+++++

```

parts	ft	increase
1	30	1.25

Example 8

Charles E Romano Jr 2-17-05
Kare & Mark - h. 2-17-05

```

+++++ (02/14/05) +++++
Solution OC-15 HEC QP-300
+++++
Total Weight Solids % Item Chemical # Identification Wet Weight Dry Weight % of dry Total Weight Wet mg/ft2 Dry mg/ft2 Total mg/ft2
-----
105.86 2.84 -1- HEC QP-300 102.85 3.006 100.00% 105.86 3421.13 100.00 3521.13
44.14 0.00 -2- Water 44.14 0.000 0.00% 44.14 1468.20 0.00 1468.20
0.00 10.00 -3- APG 325N 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
0.00 10.00 -3- Olin 10G 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
-----
150.00 146.99 3.01 100.00% 150.00 4889.33 100.00 4989.33
=====
The calculated dry weight of the batch is: 3.01 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 30.06 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.24
Add 50g for each hopper

```

parts	ft	increase
1	30	1.25

Control Example 7

105.86
44.14

Charles E Romanos Jr 2-17-05
12... e m 1. /

OC-16 K-100LV.xls
2/16/2005 11:39 AM

```

+++++ (02/14/05) +++++
Solution OC-17 A15LV
+++++
Total Weight 128.34 2.34 -1- A15LV 125.34 3.003 100.00% 128.34 4173.50 100.00 4273.50
21.66 0.00 -2- Water 21.66 0.000 0.00% 21.66 721.27 0.00 721.27
0.00 10.00 -3- APG 325N 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
0.00 10.00 -3- Olin 10G 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
-----
150.00
=====
The calculated dry weight of the batch is: 3.00 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 30.03 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.36
Add 50g for each hopper

```

Condres 1 Example 9

parts	ft	increase
1	30	1.25

Charles E Roman & 2-17-05
Karen & Mark Roman 2-17-05

```

+++++ (02/14/05) ++++++
Solution OC-18 Carbose LT-30
+++++
Total Weight 9.59 31.20
140.41 0.00
0.00 10.00
0.00 10.00
+++++
Support Type:
Coated Width: 4.50 inches
Support Width: 5.00 inches
+++++
Total Weight 9.59 31.20
140.41 0.00
0.00 10.00
0.00 10.00
+++++
Chemical Identification Wet Weight Dry Weight % of dry Total Weight
-1- Carbose LT-30 6.60 2.993 100.00% 9.59
-2- Water 140.41 0.000 0.00% 140.41
-3- APG 325N 0.00 0.000 0.00% 0.00
-3- Olin 10G 0.00 0.000 0.00% 0.00
+++++
Wet mg/ft2 220.51 100.00 320.51
Dry mg/ft2 4691.05 0.00 4691.05
Total mg/ft2 0.00 0.00 0.00
+++++
150.00
147.01 2.99 100.0% 150.00 4911.56 100.00 5011.56
=====
The calculated dry weight of the batch is: 2.99 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 29.93 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.71
Add 50g for each hopper
+++++

```

Control Example 10

parts	ft	increase
1	30	1.25

Charles E Romano Jr 2-17-05
Karen E Markesky 2-17-05

```

+++++ (02/14/05) +++++
Solution OC-19 GH-23 PVA
+++++
Total Weight Solids % Item Chemical Identification Wet Weight Dry Weight % of dry Total Weight Wet mg/ft2 Dry mg/ft2 Total mg/ft2
-----
38.25 10.00 10.00 -1- GH-23 PVA 26.96 2.995 100.00% 29.95 900.00 100.00 1000.00
53.4 0.00 0.00 -2- Water 120.05 0.000 0.00% 120.05 4007.57 0.00 4007.57
0.00 10.00 0.00 -3- APG 325N 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
0.00 10.00 0.00 -3- Olin 10G 0.00 0.000 0.00% 0.00 0.00 0.00 0.00
-----
150.00 147.00 3.00 100.0% 150.00 4907.57 100.00 5007.57
=====
The calculated dry weight of the batch is: 3.00 g
ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005
The calculated solids of this batch is: 2.00%
ENTER THE DRY COVERAGE: 0.100 grams per ft2
The calculated yield of this batch is: 29.95 ft²
COATED FOOTAGE: 37.50
TOTAL SOLUTION NEEDED: 155.63
Add 50g for each hopper

```

Control Example 11

parts	ft	increase
1	30	1.25

Charles E. Romano Jr 2-17-05
Karen E. Maskosky 2-17-05

for Feb 18 SCS

(02/14/05)

Solution OC-20 WO-320 PVA

Total Weight	% Solids	Item #	Chemical Identification	Wet Weight	Dry Weight	% of dry	Total Weight	Wet mg/ft2	Dry mg/ft2	Total mg/ft2
46.17	6.50	-1-	WO-320 PVA	43.17	3.001	100.00%	46.17	1438.46	100.00	1538.46
103.83	0.00	-2-	Water	103.83	0.000	0.00%	103.83	3459.65	0.00	3459.65
0.00	10.00	-3-	APG 325N	0.00	0.000	0.00%	0.00	0.00	0.00	0.00
0.00	10.00	-3-	Olin 10G	0.00	0.000	0.00%	0.00	0.00	0.00	0.00

150.00

The calculated dry weight of the batch is:

3.00 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION->

1.005

The calculated solids of this batch is:

2.00%

ENTER THE DRY COVERAGE:

0.100 grams per ft2

The calculated yield of this batch is:

30.01 ft²

COATED FOOTAGE:

37.50

TOTAL SOLUTION NEEDED:

155.43

Add 50g for each hopper

Control Example 12

parts	ft	increase
1	30	1.25

Charles E. Evans Jr 2-17-05
Karen E. Maskesky 2-17-05

50716 1
Bottle 2

(1) Gel 7869
(2) DI
(3) 5-100 matte

Support Type:
Coated Width: 4.50 inches
Support Width: 5.00 inches

(02/14/05)

Solution BL-01 Succinylated Gelatin

Total Weight	% Solids	Item #	Chemical Identification	Wet Weight	Dry Weight	% of dry	Total Weight	Wet mg/ft2	Dry mg/ft2	Total mg/ft2
298.28	90.00	-1-	K&K Gel 7869	29.83	268.452	99.40%	298.28	110.44	993.97	1104.41
2397.65	0.00	-2-	Water	2397.65	0.000	0.00%	2397.65	8877.55	0.00	8877.55
4.07	40.00	-3-	S-100 matte be	2.44	1.628	0.60%	4.07	9.04	6.03	15.07
2700.00				2429.92	270.08	100.0%	2700.00	8997.04	1000.00	9997.04

298.28
2397.65
4.07

The calculated dry weight of the batch is: 270.08 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 10.00%

ENTER THE DRY COVERAGE:

1.000 grams per ft2

The calculated yield of this batch is:

270.08 ft²

COATED FOOTAGE:

500.00

TOTAL SOLUTION NEEDED:

2861.67

1 Add 50g for each hopper

parts	ft	increase
1	400	1.25

PN. 2. 5 Roman 2 2-17-05

```

+++++ (02/14/05) ++++++
Solution IL-01 Elv 52-22 + Wcb-232
+++++
Total Weight 564.64 9.54 -1- Elv 52-22 510.77 53.867 77.00% 564.64 1095.12 115.49 1210.61
53.65 30.00 -2- Wcb-232 37.55 16.095 23.00% 53.65 80.52 34.51 115.02
781.71 0.00 -3- Water 781.71 0.000 0.00% 781.71 1676.02 0.00 1676.02
-----
1400.00
=====
The calculated dry weight of the batch is: 69.96 g

ENTER SPECIFIC GRAVITY OF THE SOLUTION-> 1.005

The calculated solids of this batch is: 5.00%

ENTER THE DRY COVERAGE: 0.150 grams per ft2
The calculated yield of this batch is: 466.41 ft2
COATED FOOTAGE: 750.00

TOTAL SOLUTION NEEDED: 1316.32 Add 50g for each hopper
+++++

```

parts	ft	increase
1	600	1.25

Charles E Romano 2-17-05
Lan. & M. L. /

Charles E. Romano Jr. 2-17-05

10% PVA Stock Solutions

Feb 16, 2005

1000 gm (10gm solid)

Z320 PVA
✓ 103.52g 96.6% Z320, lot 46902
✓ ↓ 896.48g DI water
1000g Δ 24 Sept 2004 data sheet

1000g Z210 PVA

✓ 104.38g Z210, lot 43903
✓ ↓ 895.62g DI water
1000g Δ 24 Sept 2004 data sheet

Charles E Romano Jr 2-17-05
Karen E Maskeasky 2-17-05